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WADC TECHNICAL REPORT 54-17

JOINING OF MOLYBDENUM

W. N. PLATTE

WESTINGHOUSE ELECTRIC CORPORATION

AUGUST 1954

Statement A
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WRIGHT AIR DEVELOPMENT CENTER

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WADC TECHNICAL REPORT 54-17

JOINING OF MOLYBDENUM

W. N. PLATTE

WESTINGHOUSE ELECTRIC CORPORATION

AUGUST 1954

MATERIALS LABORATORY
CONTRACT No. AF 18(600)114
RDO No. 446-48 (L-G)

WRIGHT AIR DEVELOPMENT CENTER
AIR RESEARCH AND DEVELOPMENT COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

Carpenter Litho & Prtg. Co., Springfield, O.
200 - 6 January 1955

FOREWORD

This report was prepared by the Westinghouse Electric Corporation under USAF Contract No. AF 18(600)114. The contract was initiated under Research and Development Order No. 446-48(L-G), "Research On Methods Of Producing Sound Ductile Joints Molybdenum", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Lt T. Hikido acting as project engineer.

ABSTRACT

By the use of a closed chamber and a continuous flow of gas through this chamber a controllable welding atmosphere has been provided for experimental work. The atmosphere may be varied from 100% argon, 99.95% pure, to 100% O_2, N_2, or any combination of these or other gases. The chamber provides a fresh gas shield in an atmosphere of the same composition as the shield.

Using the controlled atmosphere chamber, welds in carbon deoxidized arc cast molybdenum are shown to be subject to hot short cracking when the oxygen content in the argon atmosphere around the arc exceeds 0.2%. The ductility of these welds was drastically reduced by the presence of more than 0.05% oxygen in the welding atmosphere.

Oxygen in sintered molybdenum is shown to produce porosity and hot short cracking. However, crack and porosity free welds have been produced by using deoxidizing agents in sintered molybdenum.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

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INTRODUCTION

The problem of the determination of a method for producing, in molybdenum, sound welds having engineering ductility is complex in its nature. This multiple problem may be resolved into three basic divisions: first, how can existing material be improved so that sound welds are produced, second, what welding techniques are required, and, third, what test means are to be used to determine the degree of ductility obtainable. All three problems must be approached simultaneously.

In the past, welds in molybdenum have been made using a number of different welding techniques. Both electrical resistance and arc welding methods have been applied. A number of inherent disadvantages exist in each of these methods. Low ductility has been characteristic of all types of welds with the exception of percussive welding. Porosity and hot short cracking is prevalent in welds formed using the shielded electric arc process. Excessive electrode sticking and material contamination are characteristic of the spot and seam welding processes. The resistance butt and flash butt welding processes both require shielding during welding to prevent oxidization and are limited in application to particular types and sizes of joints. The last limitation is especially applicable to the use of percussive welding which, while it produces fairly ductile welds, is limited to small sections; also, commercial equipment is not available. Of the welding methods available, the inert gas arc is the most versatile; it can be used to produce most types of joints. It was because of this wide field of potential usefulness that work was undertaken in using the inert gas shielded tungsten arc process for welding molybdenum.

Welds made using any of the fusion welding processes are subject to the same basic problems inherent in any cast structure. Large dendritic grains and ingot characteristics are present. Gases trapped or combined in the original material or dissolved in the molten puddle during welding tend to be released either while the metal is molten or on cooling and this may cause weld porosity. Impurities segregating in the grain boundaries may cause either hot short cracking or low ductility at room temperature. Welds in molybdenum involve no exceptions to these phenomena. Materials having a body centered cubic type lattice structure are subject to a cleavage type failure along the 100 plane.[1] Since molybdenum has a body centered cubic lattice, a similar cleavage should be expected. Reports by other investigators have shown that molybdenum is sensitive to impurities[2] and large grain size.

The principal problem in producing sound, ductile welds in molybdenum is that of improvement of the material in and adjacent to the weld area. The original material to be welded must have a high degree of purity with respect to such agents which cause porosity and lowered ductility. This degree of purity must be maintained during the welding process and subsequent cooling.

Weld beads made in molybdenum using the inert gas shielded arc process prior to the start of these investigations showed that commercially available sintered molybdenum was inferior to the arc cast carbon deoxidized material. X-ray photographs of arc welds made in a chamber containing pure helium are shown in Figures 1 and 2. The gross cracking and porosity demonstrated in Figure 1 is representative of commercial sintered molybdenum as currently produced. Only one small crater crack may be observed in the arc cast carbon deoxidized material in Figure 2. On the basis of tests of this

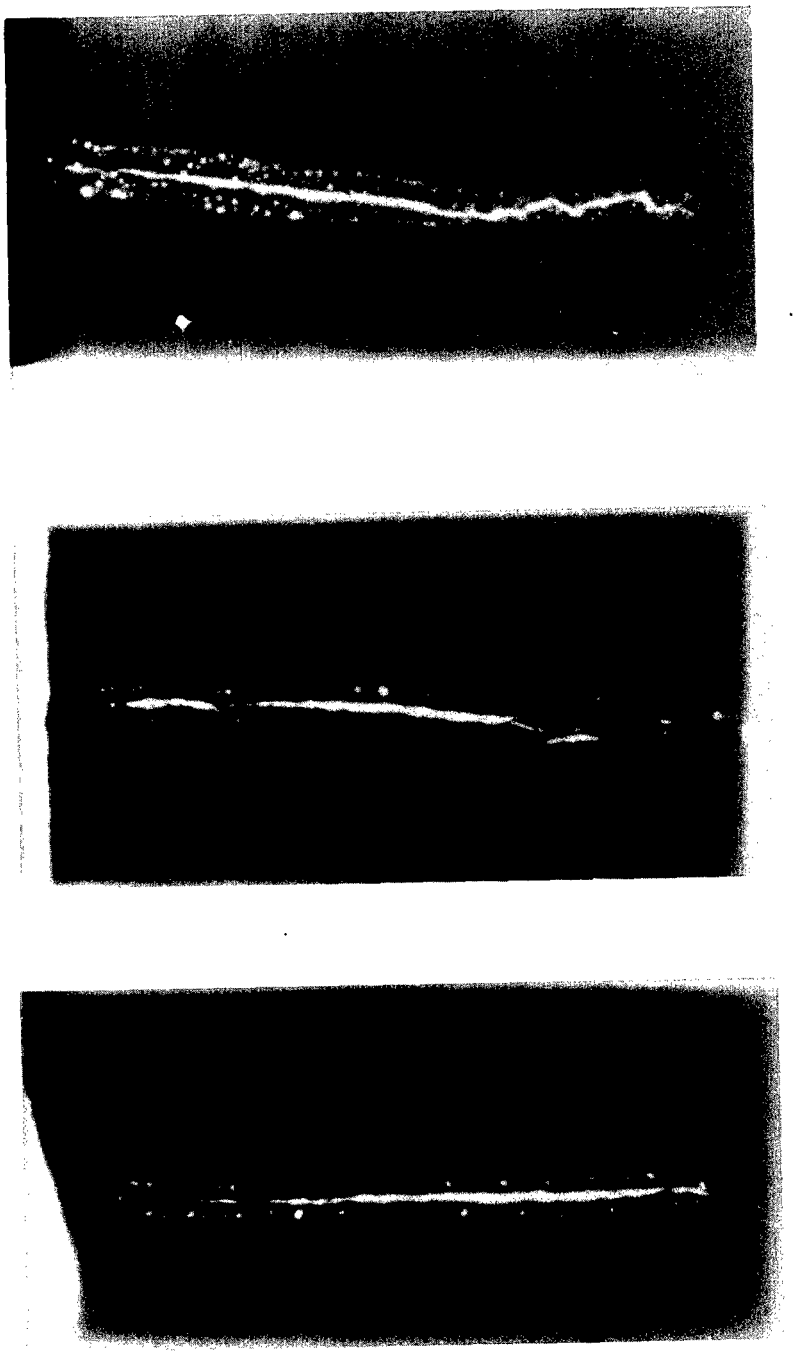


FIG.1 X-RAY PHOTOGRAPHS OF WELD BEADS ON NON DEOXIDIZED SINTERED MOLYBDENUM WELDED IN A HE. ATMOSPHERE.

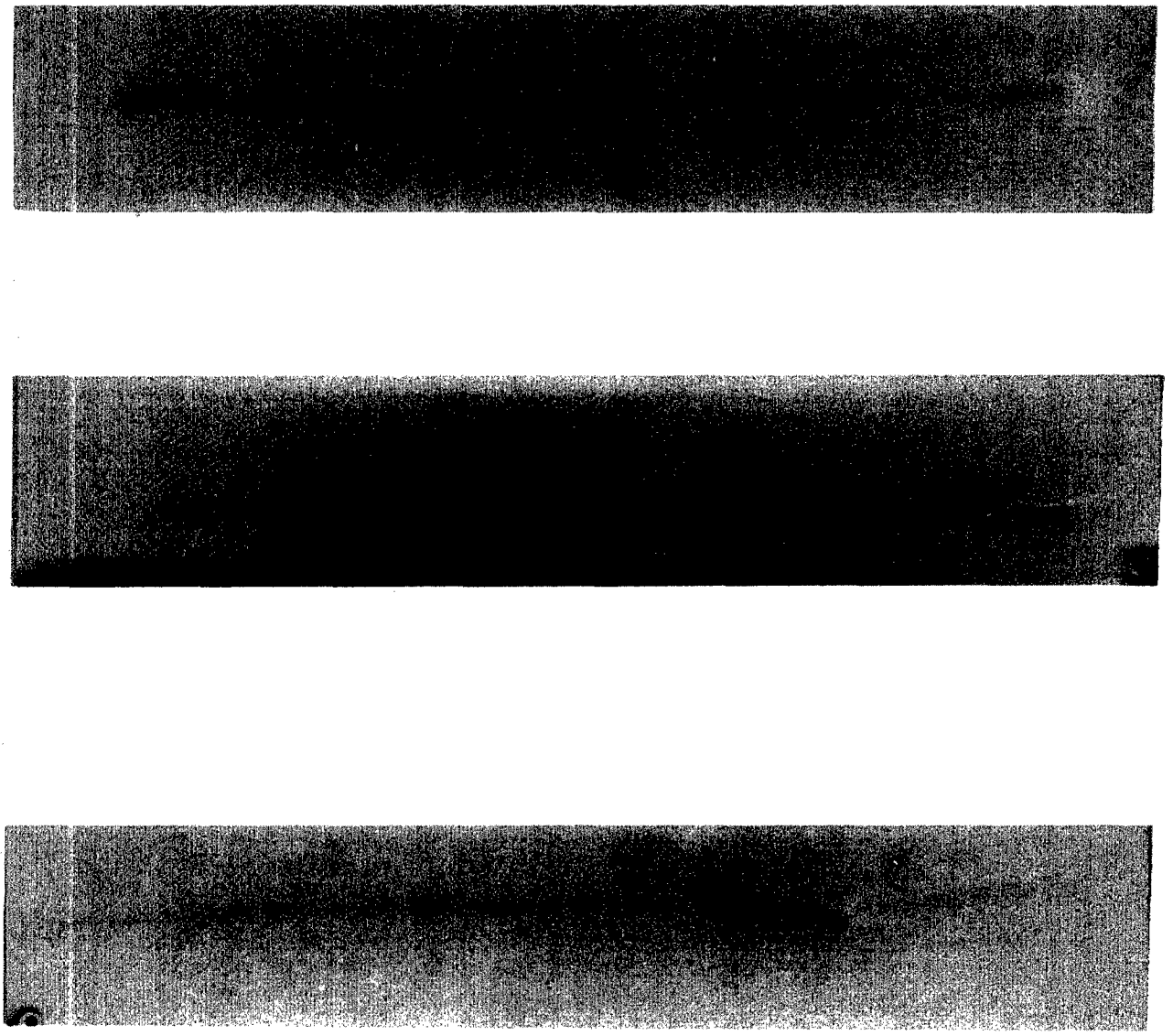


FIG. 2 X-RAY PHOTOGRAPHS OF WELD BEADS ON C. DEOXIDIZED ARC CAST MOLYBDENUM MADE IN A PURE HE. ATMOSPHERE.

I - MATERIAL

The material used in the welding tests consisted of carbon deoxidized arc cast molybdenum obtained from Climax Molybdenum Corp. and deoxidized vacuum sintered materials made up in the Westinghouse Research Laboratories. The vacuum sintered material was made up as a part of the material improvement program.

The arc cast molybdenum from Climax Molybdenum Corp. contained 0.06% C, 0.0018% O_2 and 0.003% N_2 by check analysis. This material was received in the form of sheet bars machined to 1-1/2 x 4 x 6 inches. These sheet bars were reduced to .065 in. x 7 in. sheet. A complete listing of the fabrication procedure is given in Table I. The rolling schedule was based upon data obtained previously in other investigations.[3] The data, shown in Figure 3, served as a basis for the rolling schedule. The basic principle of this procedure requires that for the amount of reduction used, the time and reheating temperatures must be so chosen that recrystallization does not occur during the rolling process. This rolling procedure has been found to produce a fine fibered structure in the worked condition which can be recrystallized to a fine equiaxed structure. The microstructures of the arc cast molybdenum during several stages of rolling are shown in Figures 4-11. These photomicrographs demonstrate the adherence to the rolling procedures derived from Figure 3. Cross sections of two billets, 993-B1-2 and 993-B2-2, are shown in Figures 4 and 5 at 1.5X. Portions of these same billets are shown in Figures 6 and 7 at 100X. The billets were annealed one hour at 1350-1400°C in hydrogen to produce a uniform equiaxed structure. The microstructure of the two billets are shown at 100X after annealing in Figures 8 and 9. The structure of the molybdenum is shown in figure 10 after final rolling. This

TABLE I

FABRICATION OF ARC CAST MOLYBDENUM SHEET

1. Consolidation: Arc cast by Climax Molybdenum Co.

2. Extrusion: (a) Heated to 1260°C, extruded from 6-1/2" dia. to 4" dia.

   (b) Recrystallized by heating 1 hour at 1425°C.

3. Forging: (a) Forged to 1-3/4 x 4-3/4 at 1260-1310°C.

   (b) Machined to 1-1/2 x 4" and cut into 6" x 8" sheet bars.

   (c) Annealed for 1 hour at 1350-1400°C.

4. Intermediate Rolling:

   (a) Slabbed to 6" x 8" x 1" by cross rolling.

Pass No.	Temperature	Reheat Time	Thickness
1	1200°C	30 min.	1-5/16"
2	1160	10	1-1/8
3	1160	10	1

   (b) Change direction of rolling 90°.

Pass No.	Temperature	Reheat Time	Thickness
4	1090°C	10 min.	7/8"
5	1070	10	5/8
6	1050	10	3/8
7	1050	10	1/4
8	1050	10	3/16
9	1050	10	1/8

5. Final Rolling:

Pass No.	Temperature	Reheat Time	Thickness
1	500°C	15 min.	.095"
2	500	5	.080
3	500	5	.067
4	---	-	.062

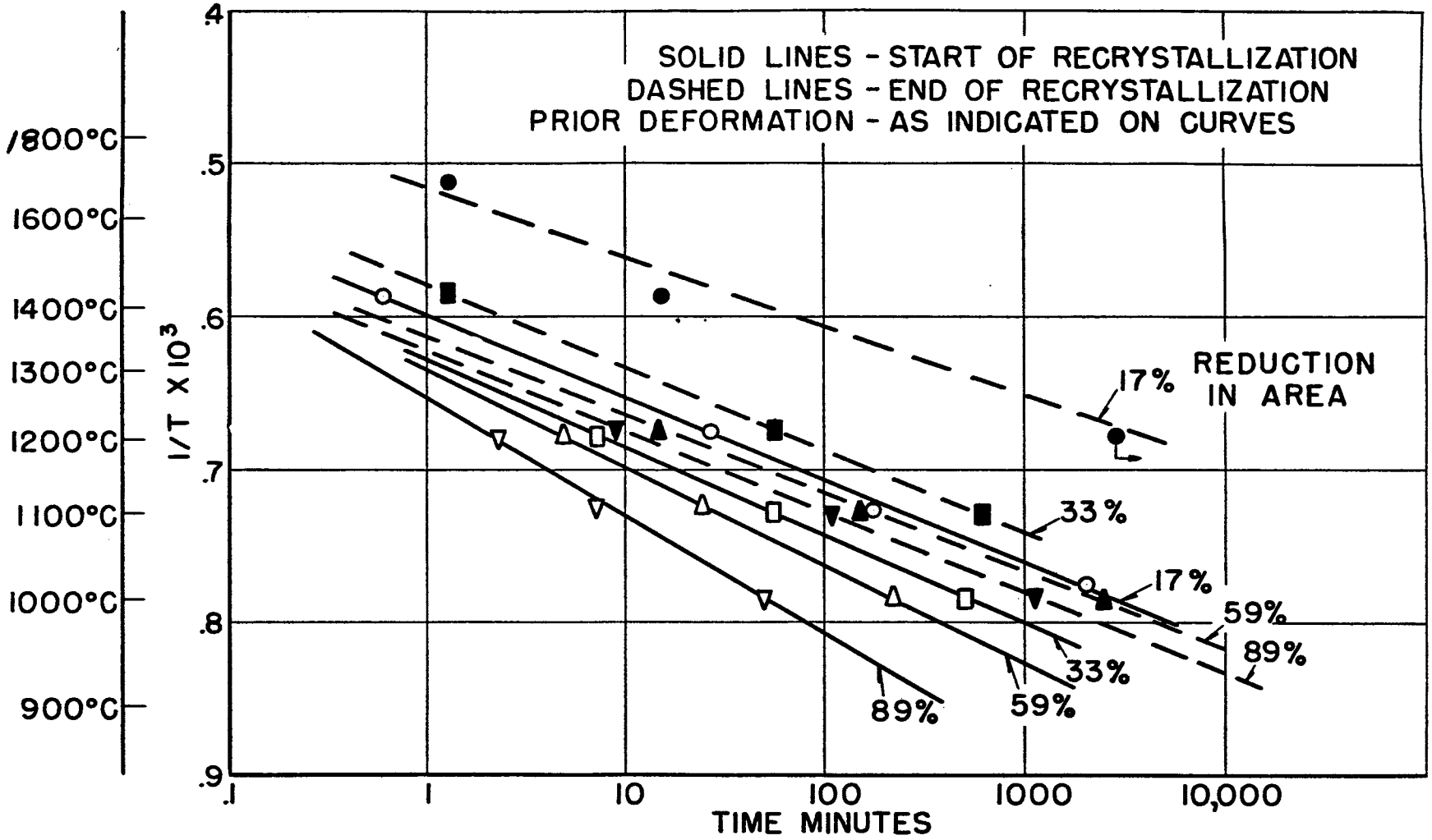


FIG. 3 - TIME-TEMPERATURE RELATIONSHIP FOR THE START AND END OF RECRYSTALLIZATION ARC-CAST MOLYBDENUM - SAMPLE CS-376.

P-53-2-1

FIG.4 MACROSTRUCTURE OF TRANSVERSE SECTION THROUGH BILLET 993-B1-2. ELECTROLYTICALLY POLISHED IN CONCENTRATED SULFURIC ACID, ETCHED IN ALKALINE K_3 $Fe(CN)_6$ SOLUTION. 1.5 X MAGNIFICATIONS

P-53-2-2

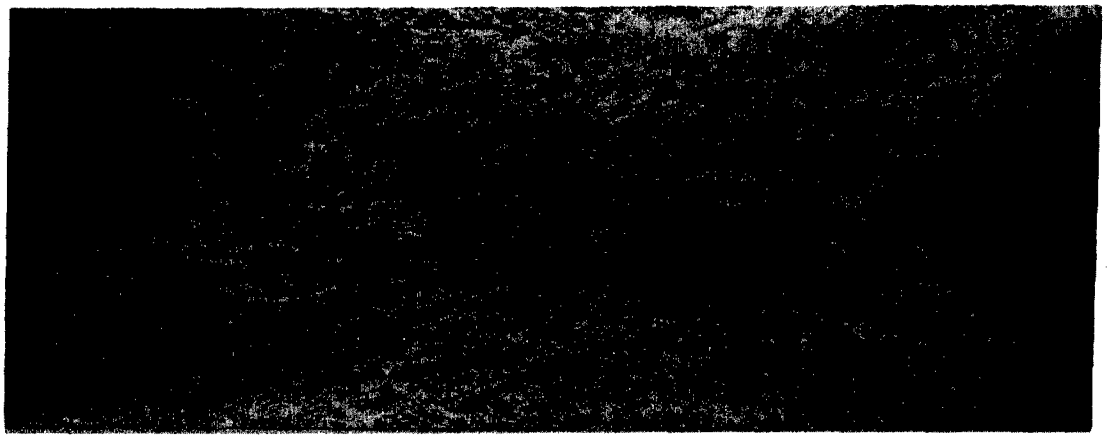


FIG. 5 MACROSTRUCTURE OF TRANSVERSE SECTION THROUGH BILLET 993-B2-2. ELECTROLYTICALLY POLISHED IN CONCENTRATED SULFURIC ACID, ETCHED IN AN ALKALINE $K_3Fe(CN)_6$ SOLUTION. 1.5 X MAGNIFICATIONS

P-53-2-3

FIG.6 TYPICAL MICROSTRUCTURE OF BILLET 993-B1-2 AS RECEIVED, 100X MAGNIFICATIONS

P-53-2-4

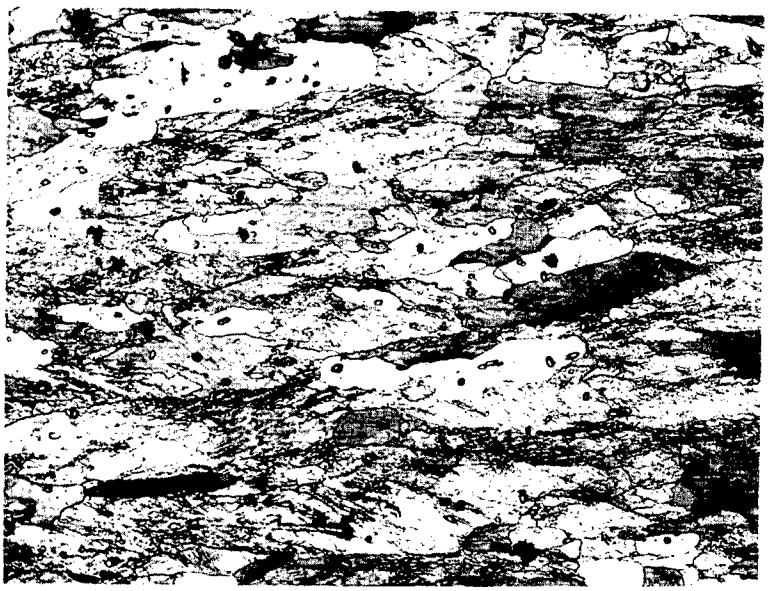


FIG.7 TYPICAL MICROSTRUCTURE OF BILLET 993-B2-2. 100 X MAGNIFICATIONS (AS RECEIVED)

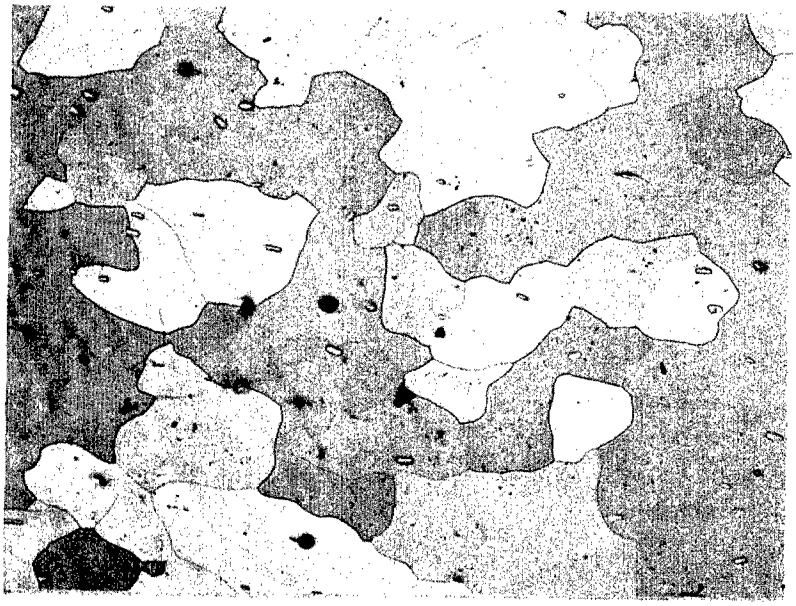


FIG. 8 MICROSTRUCTURE OF BILLET 993-B1-2 ANNEALED FOR 1 HOUR AT 1350 - 1400°C IN HYDROGEN. MAGNIFICATION 100X

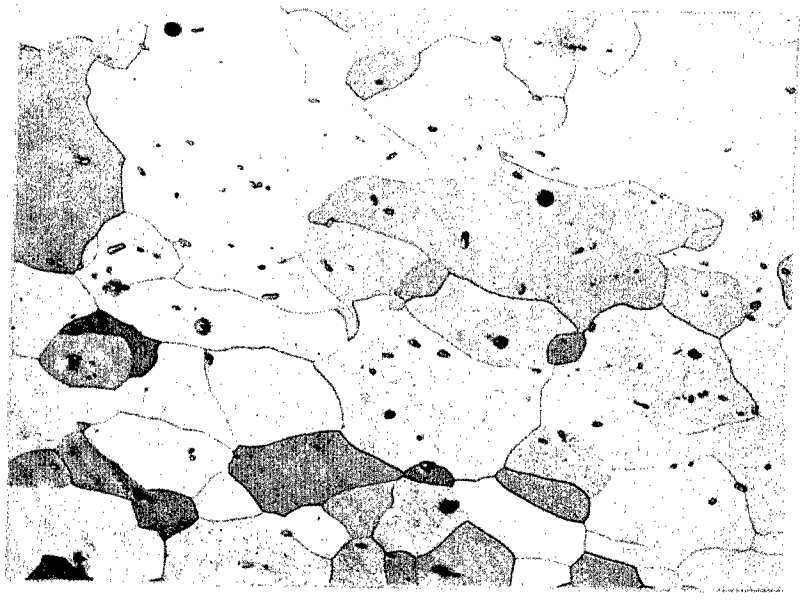


FIG. 9 MICROSTRUCTURE OF BILLET 993-B2-2 ANNEALED FOR 1 HOUR AT 1350 - 1400°C IN HYDROGEN. MAGNIFICATION 100X

P-53-197-1

FIG.10 ARC CAST CARBON DEOXIDIZED MOLYBDENUM AFTER FINAL ROLLING.

P-53-198-1

FIG.11 ARC CAST CARBON DEOXIDIZED MOLYBDENUM RECRYSTALLIZED 1300°C

figure shows the uniform fine fibered structure obtained. This fine fibered structure recrystallizes in a fine grain equiaxed structure as shown in Figure 11.

The hardness of the "as rolled" and recrystallized sheets are as follows:

"As Rolled" 320 DPH

Recrystallized at 1200°C, 1 hr, 197 DPH.

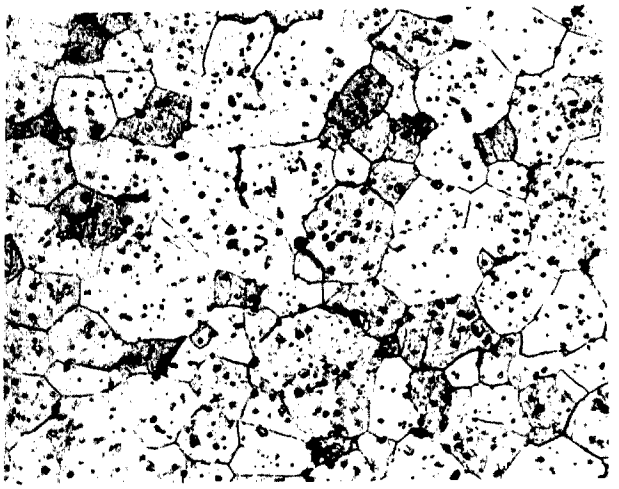
Exploratory tests were made to determine the effects of several deoxidizers on vacuum sintered molybdenum. These tests were undertaken to determine whether deoxidization, by additions to the sintered compact, would produce a sintered material suitable for welding. A group of nine sintered compacts were made up using Al, C, and Ti as deoxidizers. One control sample was made in which no deoxidizers were used. Data taken during the sintering and rolling of these compacts are given in Table II.

The rolling procedure for these sintered deoxidized samples was based on the curves in Figure 3. The material was initially rolled at 1150-1250°C. Heating was done in a hydrogen furnace atmosphere. The final passes were made at 925°C from a furnace with an EXOGAS atmosphere. The material was reduced from 0.438 in. to 0.070 in. A portion of each ingot was annealed at 1200°C for one hour. Recrystallization after annealing was complete in all but the pure molybdenum sample. It appears that a higher recrystallization temperature was required for the vacuum sintered pure molybdenum than for the deoxidized vacuum sintered material. This difference may be due to molybdenum oxides in the pure or non-deoxidized material. These oxides may act as a recrystallization inhibitor. Photomicrographs of the vacuum sintered deoxidized samples during different stages of their fabrication are shown in Figures 12-18. Chemical analysis data for the vacuum sintered deoxidized samples are given in Table II.

TABLE II

VACUUM SINTERED DEOXIDIZED MOLYBDENUM

Sample	Composition Aim	Analysis	Sintering Temperature	Sintering Time	HARDNESS DPH		
					As Sintered	As Rolled	Recrystallized
WS 146	0.1% C Bal. Mo	0.12% C Bal. Mo	1950°C	4 hrs.	143	296	176
WS 147	0.3% C Bal. Mo	-	1950°C	4 hrs.	---	303	187
WS 148	0.2% Ti Bal. Mo	0.10 Ti Bal. Mo	1950°C	4 hrs.	146	300	178
WS 149	0.5% Ti Bal. Mo	-	1950°C	4 hrs.	136	---	---
WS 150	0.2% Al Bal. Mo	0.09 Al Bal. Mo	1900°C	4 hrs.	130	298	170
WS 151	0.5% Al Bal. Mo	0.18% Al Bal. Mo	1900°C	4 hrs.	---	317	180
WS 152	Pure Mo	-	1950°C	4 hrs.	96	310	231

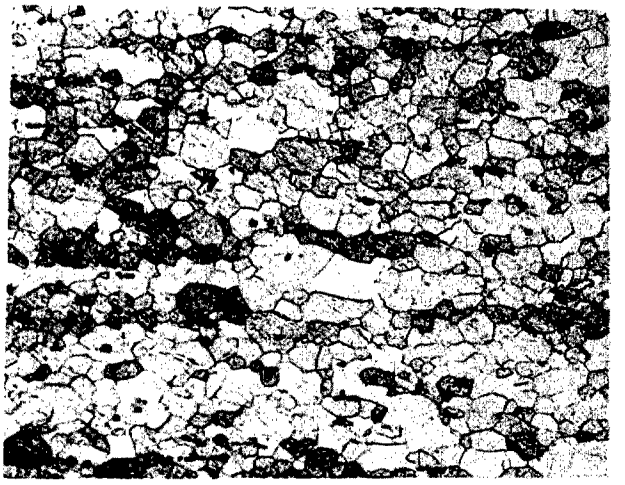


P 53-31-1

AS SINTERED

4 HRS. 1950°C

200 X



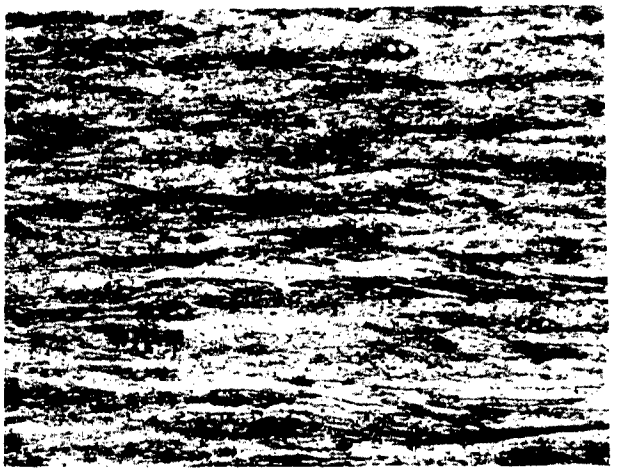
P 53-68-1

AS ROLLED

INITIAL PASSES 1150-1240°C

FINAL PASSES 925°C

85% REDUCTION 200 X



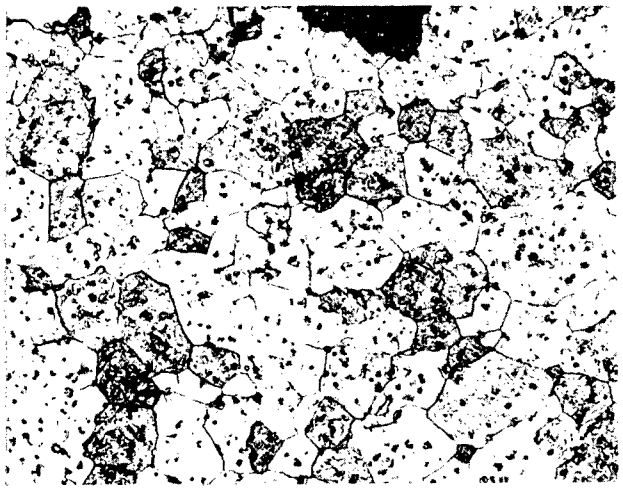
P 53-69-1

1 HR 1200°C

AFTER ROLLING

200 X

FIG.12 0.1% C BAL MO
SAMPLE WS 146



AS SINTERED
4 HRS 1950°C
200 X

P 53-31-2

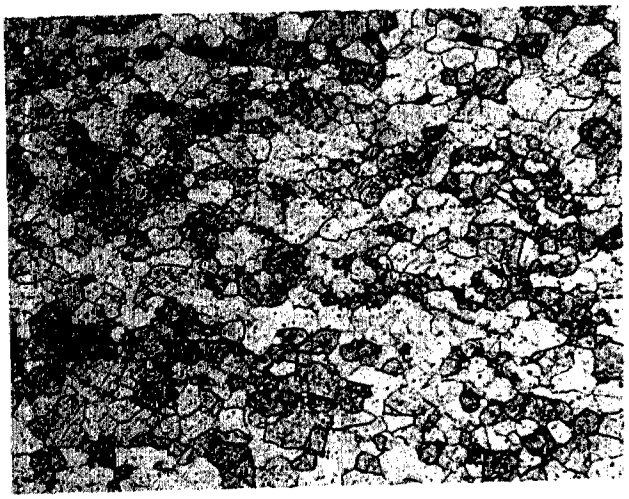
AS ROLLED
INITIAL PASSES 1150-1240°C
FINAL PASSES 925°C
85% REDUCTION 200 X

P 53-68-2

P 53-69-2

1 HR 1200°C
AFTER ROLLING
200 X

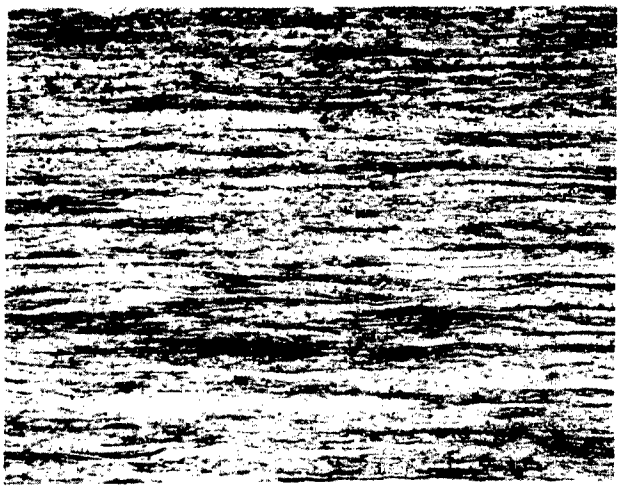
FIG.13 0.3% C BAL MO
SAMPLE WS147



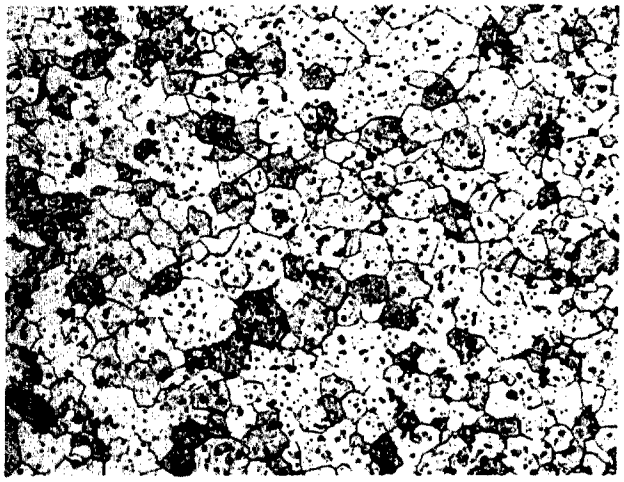
AS SINTERED
4 HRS 1950°C
200 X

P 53-31-3

AS ROLLED
INITIAL PASSES 1150-1240°C
FINAL PASSES 925°C
85% REDUCTION 200 X



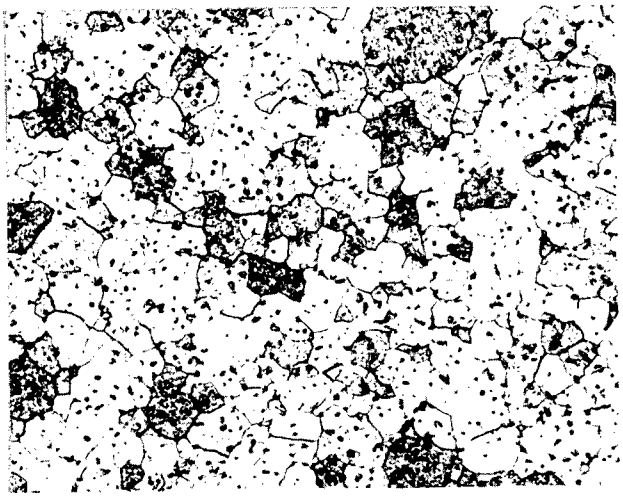
P 53-68-3



1 HR 1200°C
AFTER ROLLING
200 X

P 53-69-3

FIG. 14 0.2% T1 BAL MO
SAMPLE WS 148

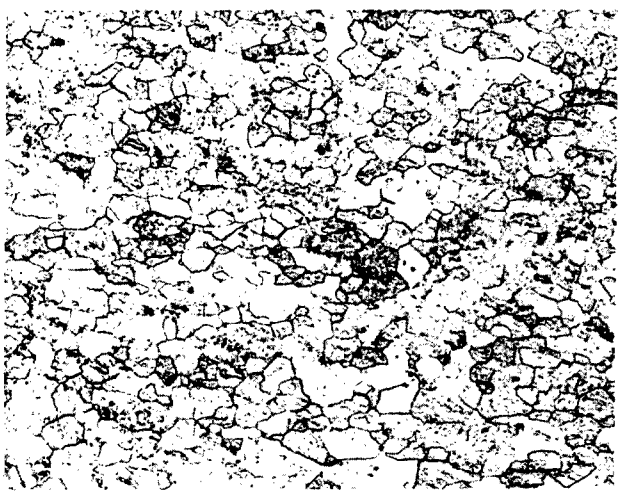


P 53-31-4

AS SINTERED
4 HRS. 1950°C
200 X

AS ROLLED
INITIAL PASSES 1150-1240°C
FINAL PASSES 925°C
85% REDUCTION 200 X

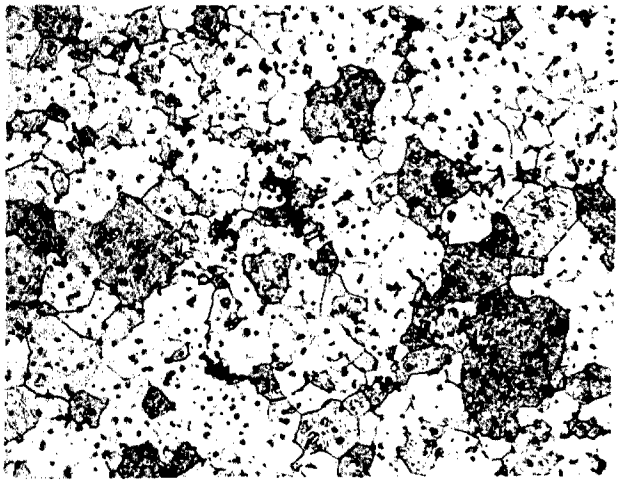
P 53-68-4



P 53-69-4

1 HR 1200°C
AFTER ROLLING
200 X

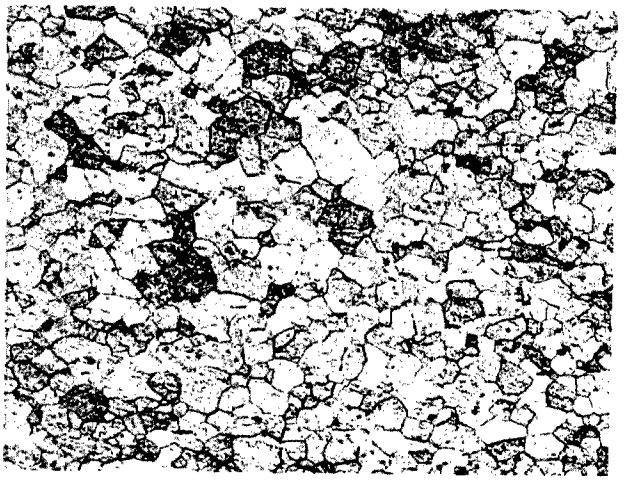
FIG. 15 0.5% T1 BAL MO
SAMPLE WS 149



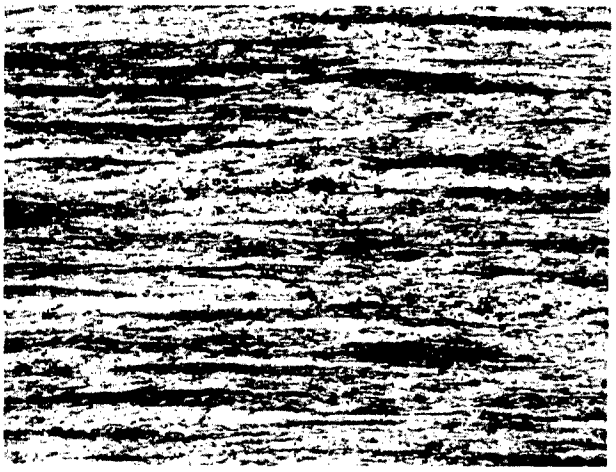
AS SINTERED
4 HRS 1950°C
200 X

P 53-31-5

AS ROLLED
INITIAL PASSES 1150-1240°C
FINAL PASSES 925°C
85% REDUCTION 200 X



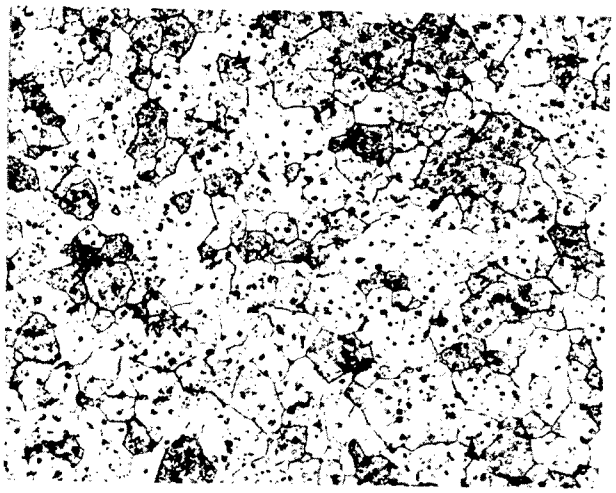
P 53-68-5



1 HR 1200°C
AFTER ROLLING
200 X

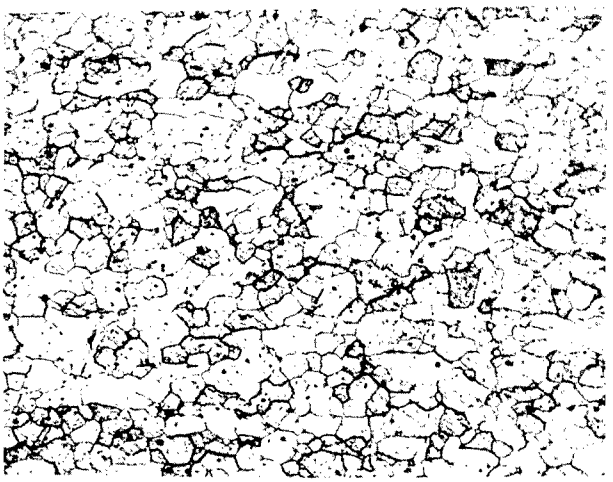
P 53-69-5

FIG.16 0.2% AL BAL MO
SAMPLE WS 150



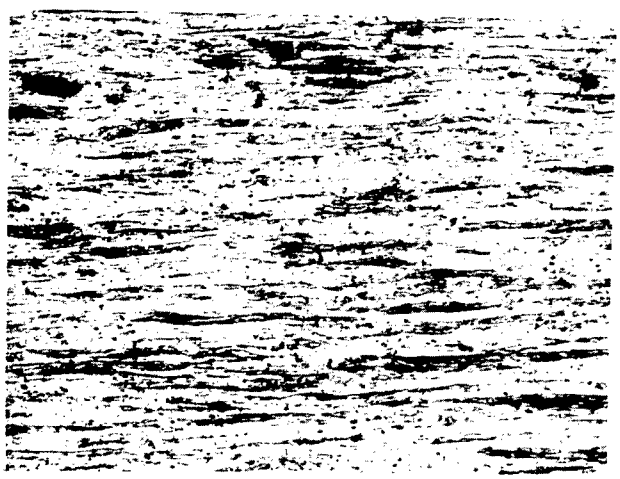
P 53-31-6

AS SINTERED
4 HRS 1900°C
200 X



P 53-68-6

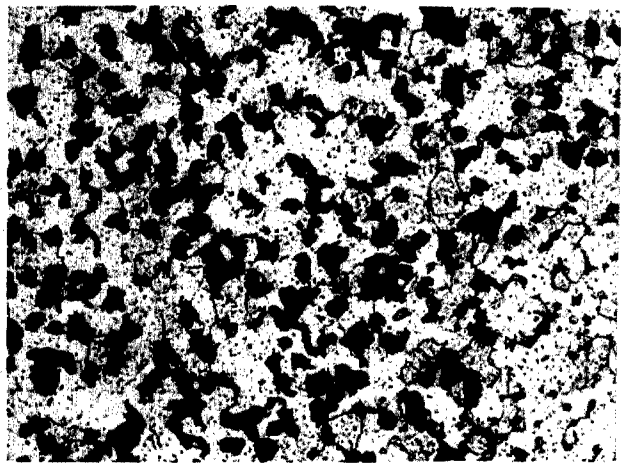
AS ROLLED
INITIAL PASSES 1150-1240°C
FINAL PASSES 925°C
85% REDUCTION 200 X



P 53-69-6

1 HR 1200°C
AFTER ROLLING
200 X

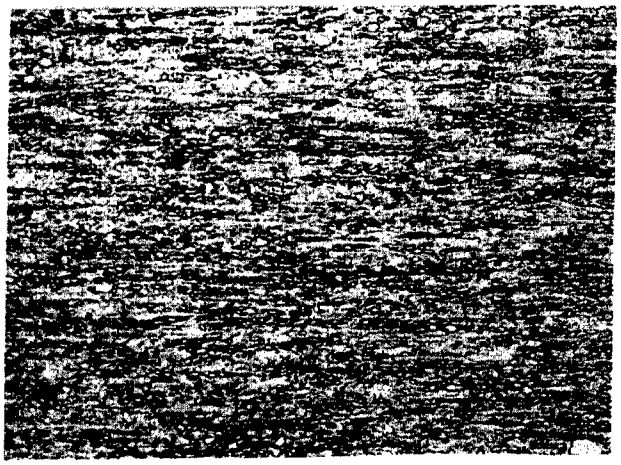
FIG. 17 0.5% AL BAL MO
SAMPLE WS 151



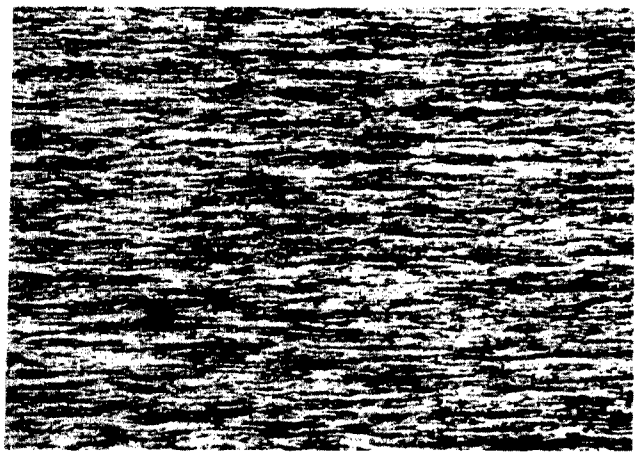
P 53-39-1

AS SINTERED
4 HRS 1950°C
200 X

AS ROLLED
INITIAL PASSES 1150-1240°C
FINAL PASSES 925°C
85% REDUCTION 200 X



P 53-68-7



P 53-69-7

1 HR 1200°C
AFTER ROLLING
200 X

FIG.18 PURE MOLYBDENUM
SAMPLE WS 152

The vacuum sintered deoxidized samples given in Table II were used as trial samples on which weld beads were made. The welding of these samples will be discussed completely under welding tests. It is sufficient at this point to say that these samples indicated an improvement over the non-deoxidized H_2 sintered material. On the basis of the data obtained from these early exploratory samples a program of material improvement through deoxidization practice has been laid out. This program involves a recheck of the effects of Ti over a range from 0.2% to 5.0%. The effects of C and Al will be re-examined and other deoxidizers such as Ta, Zr, Cb, Ca, Mg and Mn will be checked. The effects of such impurities as Fe and Ni which may occur in the powder prior to sintering will also be investigated. These impurities will be checked because spectrographic analysis of molybdenum indicated their presence in the sintered molybdenum and not in the arc cast material. Spectrographic analysis data are given in Table III. Improvement of sintered molybdenum is also planned by removal of O_2 and N_2 by high temperature vacuum heat treatment.

II - WELDING EQUIPMENT

During the early stages of the program for joining molybdenum, considerable effort was expended in the improvement and development of equipment for welding and testing. While both a welding chamber and basic test equipment were available from other investigations, it was necessary to improve and develop special devices and fixtures for the molybdenum program.

The welding chamber used to explore the effects of the purity of the welding atmosphere on the quality and ductility of molybdenum welds was

TABLE III

SPECTROGRAPHIC ANALYSIS OF SINTERED AND ARC CAST MOLYBDENUM

Element[2]	Range Estimated[1]	Climax Arc Cast Molybdenum	Westinghouse Sintered Molybdenum
Al	(0.001-0.01)	Present	Present
Ba	(0.01-0.1)	Not detected	Present
Cd	(0.001-0.05)	Present	Present
Cr	(0.001-0.05)	Barely detected	Barely detected
Cu	(0.0001-0.005)	Low	Medium
Fe	(0.005-0.1)	Low	(2-5) x higher
Pb	(0.001-0.01)	Present	Present
Mg	(0.0001-0.01)	Present	(5-10) x higher
Mn	(0.001-0.1)	Present	(3-8) x higher
Ni	(0.005-0.08)	Not detected	Present
Si	(0.005-0.05)	Present	(3-10) x higher
Sn	(0.005-0.05)	Present	Present
W	(0.05-0.5)	Barely detected	Barely detected

Notes:

1. Range compositions are only estimated since standards were not available.

2. Elements also checked but not found: Sb, Be, Bi, B, Co, Cb, Gr, Ag, Au, Na, Ta, V, Zn, Zr, As, Ti.

originally constructed for use on steel. It was necessary to modify this equipment so that consistent, high purity atmospheres could be obtained.

A photograph of the chamber and auxiliary equipment is shown in Figures 19 and 20. This chamber operates at a positive pressure of approximately 30 mm above atmospheric. Basically, the system consists of an argon shielded arc operating in an argon atmosphere. This is accomplished by causing welding grade argon to enter around the electrode during welding and after the chamber has been purged so that no residual air is present. This arrangement provides argon of maximum purity over the weld at all times and tends to remove any gases evolved from the weld metal during fusion. This system has been found to be more useful in welding molybdenum than static atmosphere chambers. The continuous purging by fresh gas has definite advantages for materials such as molybdenum which has volatile oxides. Any oxide vaporized or evolved from the metal is immediately swept away by the fresh high purity gas flowing in around the electrode. Welds made in static atmospheres in some early tests were found to have surface oxides which were the result of a gradual increase in O_2 concentration during the course of the welding tests. The dynamic atmosphere system used in the welding program prevents changes in the atmosphere due to impurity build-up.

The atmosphere supplied to the welding chamber can be varied from 100% welding grade argon 99.95% to 100% O_2 or N_2 or any combination of these or other gases. A gas mixing system is used to pre-mix the gases before they enter the welding chamber. A schematic drawing of the mixing system used for two gases is shown in Figure 21.

The welding chamber was purged by passing gas of the desired composition from the mixing system into the closed chamber. When the pressure in the

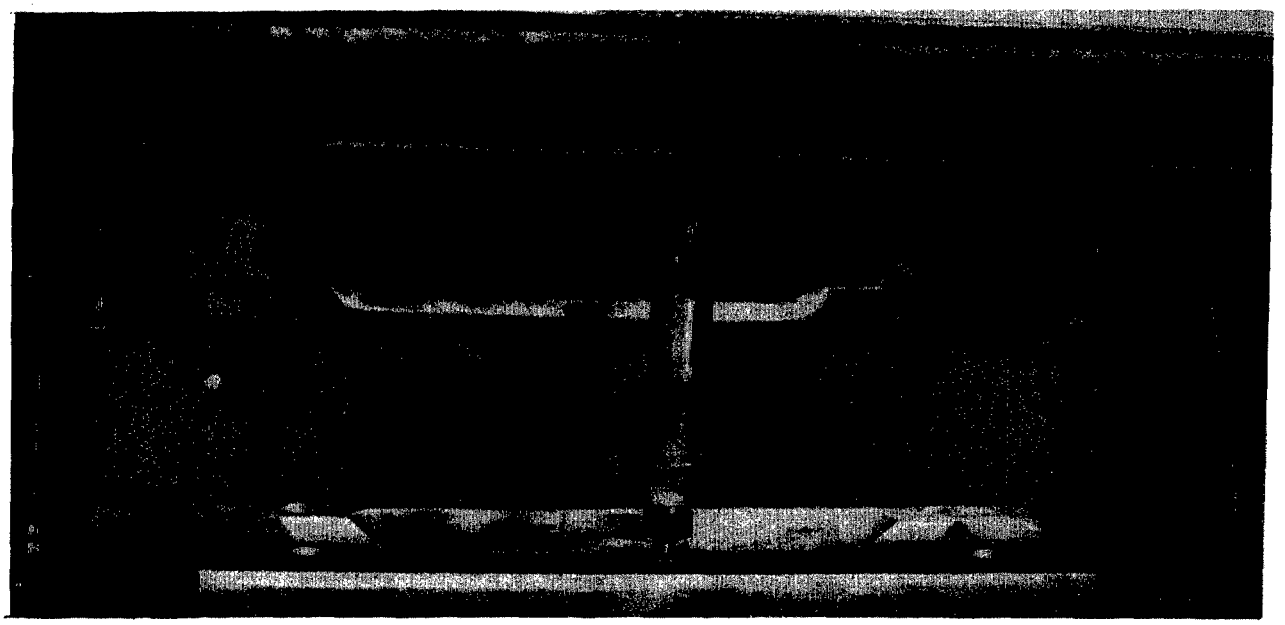


FIG.19 ARC CHAMBER

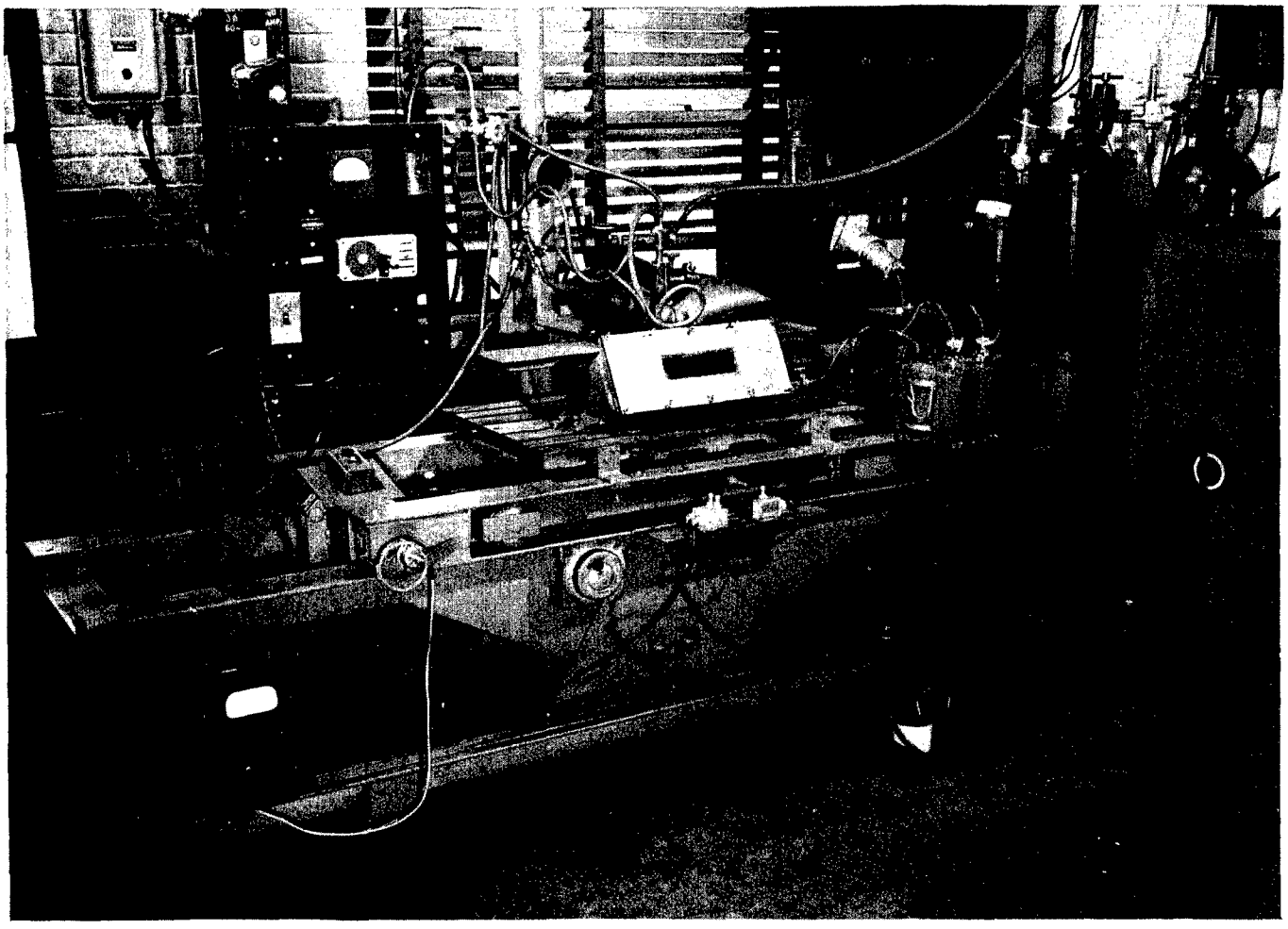


FIG. 20 ARC CHAMBER AND EQUIPMENT

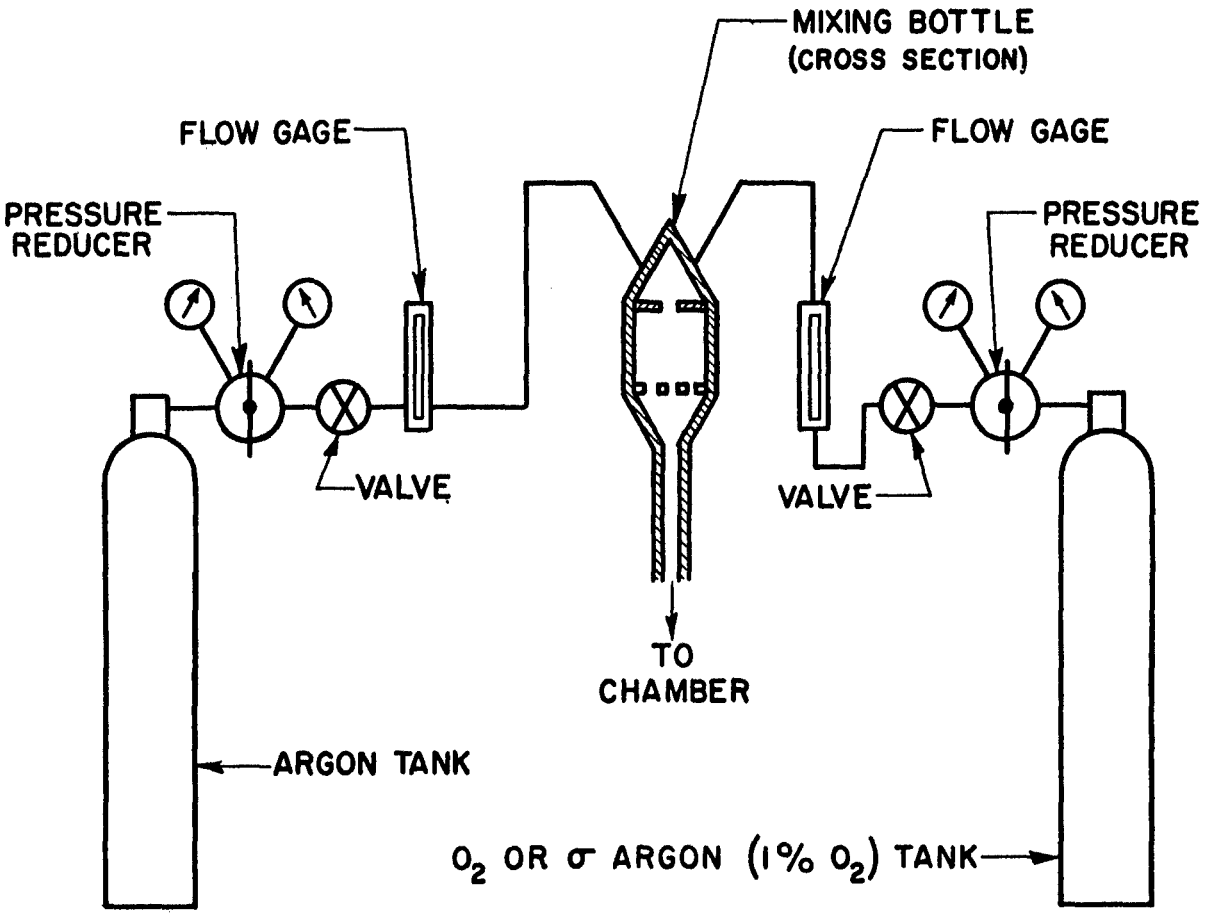


FIG. 21 - SCHEMATIC GAS MIXING SYSTEM

chamber reaches 15-20 mm of Hg, a pressure switch turns off the gas supply and starts a vacuum pump which reduces the pressure in the tank to a value slightly below atmospheric. The pump-down time is one-half minute. At the end of the pump-down period the pump is stopped and gas flow is resumed. This process is repeated 30 times. A rubber gas expansion bellows is shown in Figure 20. This expansion bellows provides a breathing action during the purge period so that after the 30 cycle purge the chamber is completely free of gases other than those supplied to it from the atmosphere mixing unit. After the final purging cycle has been completed the vacuum pump is stopped and when the pressure in the chamber reaches 30 mm of Hg, a separate exhaust is automatically opened. Restrictions in the exhaust line are adjusted so that with a continuous flow of 27.5 cu ft/hr of gas the pressure in the chamber will be held at approximately 30 mm of Hg above atmospheric pressure. The purge cycle and gas flow were established in an arbitrary manner and have been found to provide an atmosphere equivalent to the tank purity of the gases used.

The atmosphere of the chamber can be sampled by means of a sample bottle which draws gas from a portion of the exhaust line of the chamber. Provision was made so that this line was purged with the chamber so that the gas in the sampling line is the same as that in the chamber. Gas sampling was done by Hg displacement during the tests discussed in this report. However, this system will be replaced by a sampling system in which the sample bottle becomes a physical part of the exhaust line rather than an appendage. It is planned that the new system will simplify and improve the ease and quality of sampling.

Samples of welding chamber atmospheres were analyzed by means of the mass spectrometer.

Welding was accomplished by means of a direct current tungsten arc. A Westinghouse RA rectifier welder, Style 1458540, Serial No. 153818, supplied the welding current. Straight polarity was used which made the molybdenum plate the anode. This places the major portion of the heat from the arc on the molybdenum sheet. The welding current was maintained at 180 amps for the .062 in. molybdenum sheet. A Westinghouse spark gap oscillator, Style 1547011-A, Serial No. 100, was used to provide a high frequency spark for arc initiation. An Esterline Angus recorder was used to record all welding current and voltage data.

The tungsten electrode used was 3/32 in. in diameter and the distance between the electrode and the plate was 3/32 in. Movement of the electrode with respect to the plate is entirely mechanical and manual manipulation of the arc during welding is eliminated. The electrode remains stationary while the chamber to which the weld sample is fastened moves relative to the electrode. The movement, or welding speed, is controlled by means of a Graham variable speed transmission. This drive unit was originally intended for use at higher travel speeds and some difficulty has been found in duplication of welding speeds. A new drive unit has been designed and suitable equipment ordered to correct this deficiency. A drawing of the new drive system is shown in Figure 22.

Measurement of the travel speed also presents difficulties. Tachometers for speeds under 10 RPM are not readily obtainable. A device was constructed using a gear train and a small motor operating as a generator. The emf produced has been calibrated as a function of travel speed. This device serves as an indicator only. The true travel speed is obtained by dividing the arc time as given by the current record by the distance traveled.

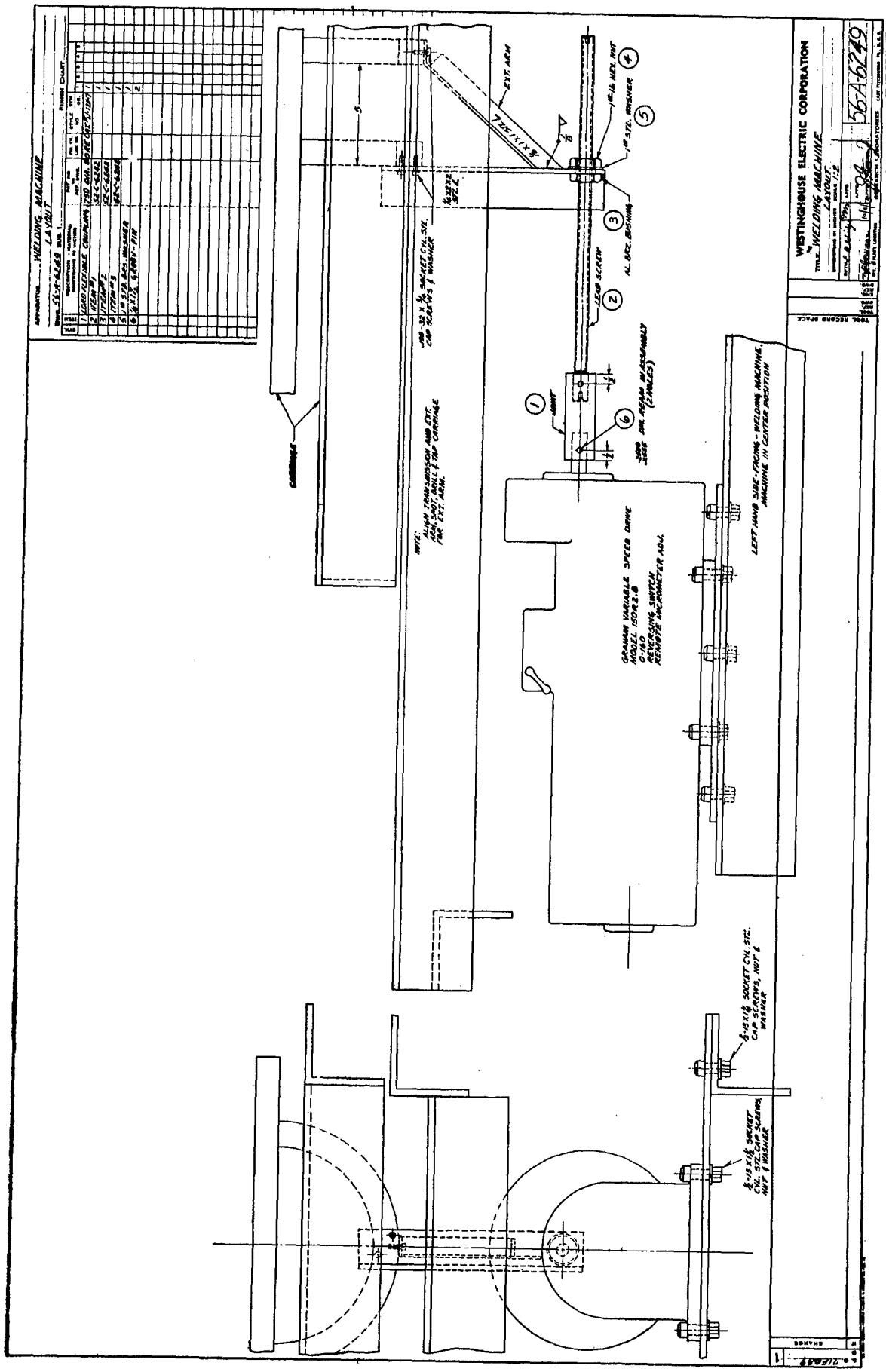


FIGURE 22 - WELDING MACHINE LAYOUT

Acceleration occurs prior to arc initiation and deceleration of the system occurs after the arc is out.

III - TESTING EQUIPMENT

Guided bend tests and tensile tests were used in these investigations to determine ductility. Testing equipment was available at the Westinghouse Research Laboratories, but it was necessary to design and construct special fixtures for testing weld samples. Due to the restrictions on weld sample size and the expense of the material, it was decided to use specimens .062 in. x 1/4 in. x 1.125 in. and .062 in. x 1/4 in. x 2 in. Detailed drawings of the specimens and their location in the weld sample are shown in Figures 23-25.

The fixture for guided bend testing of weld beads is shown in Figure 26. The weld is placed in this fixture as shown in the assembly drawing. The weld bead is located opposite the ram with the top of the bead facing away from the ram. This causes a maximum tensile stress on the outer fibers of the weld.

Tensile test fixtures were also designed and constructed for existing equipment. A drawing of these test fixtures are shown in Figure 27.

Testing at temperatures above ambient was accomplished by means of a conventional tube type furnace with a maximum temperature gradient of $\pm 5°F$ over the test area. This furnace could be controlled to $\pm 5°F$. Testing below ambient temperatures was accomplished by means of a special low temperature chamber shown in Figure 28. The chamber is shown with the bend test fixture in position. The temperature of the specimen is controlled by means of a thermocouple placed in the center of the bending ram at its point of contact

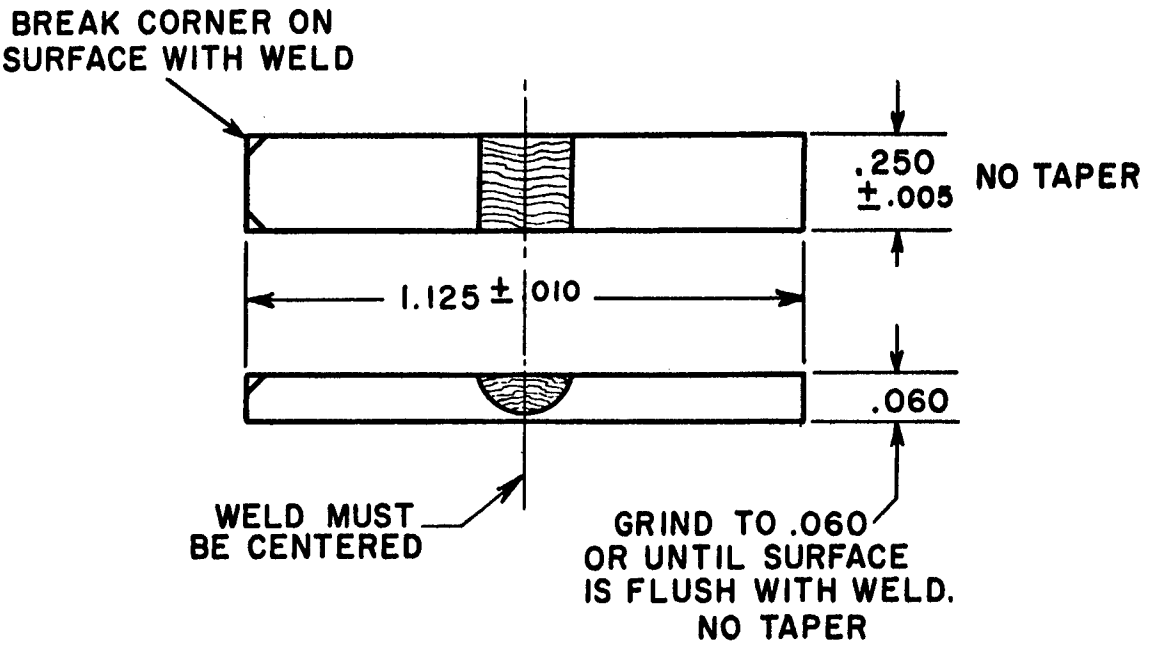


FIG. 23 - BEND TEST SPECIMEN

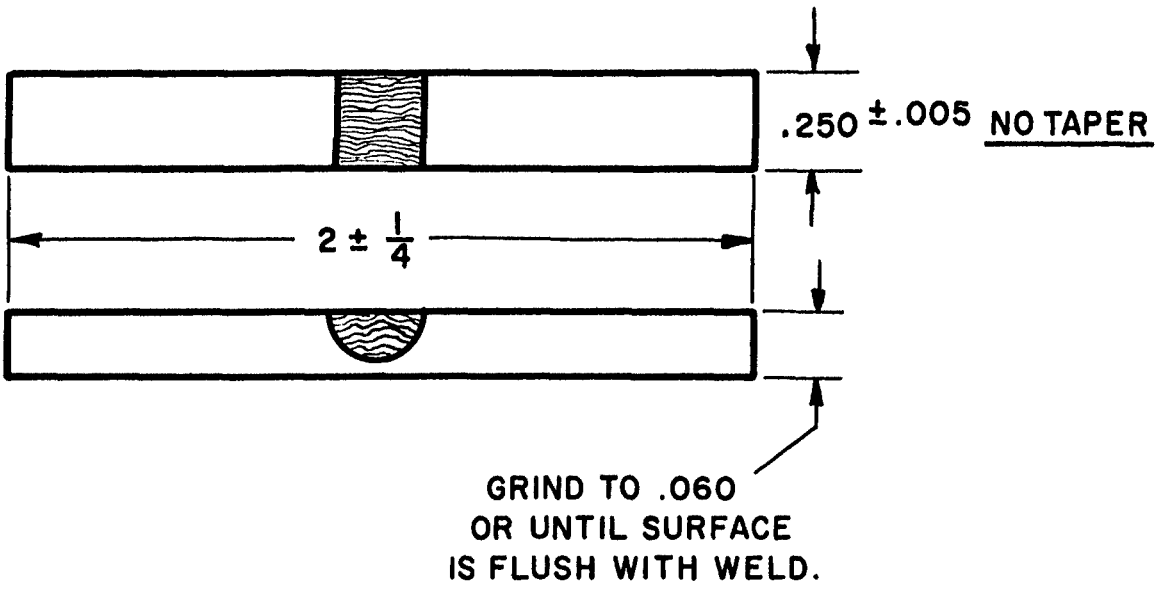


FIG. 24 - TENSILE SPECIMEN

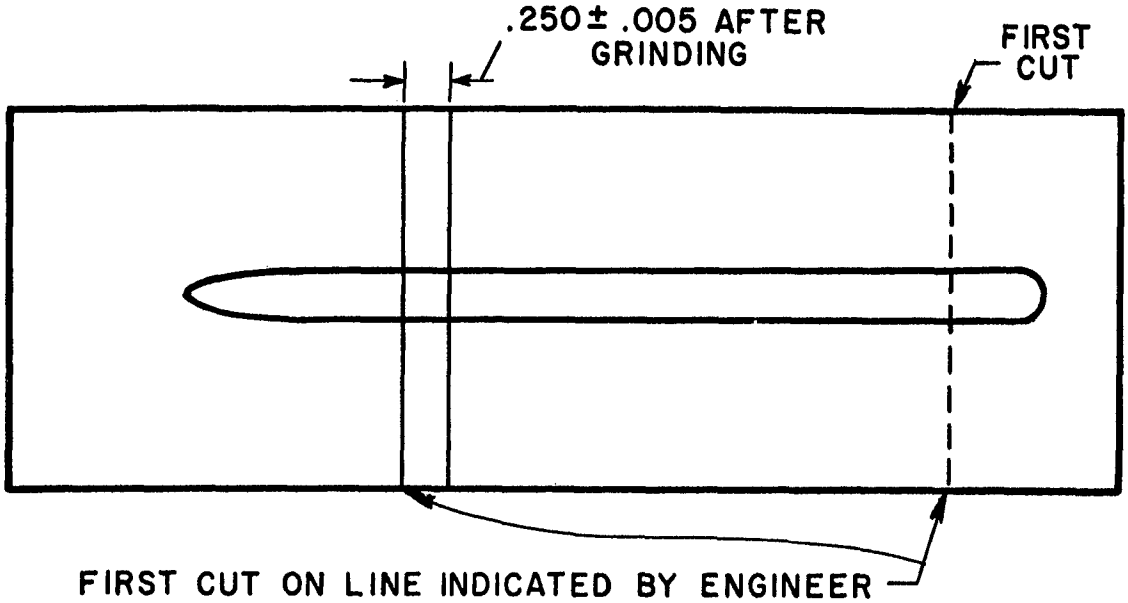


FIG. 25 - WELD SPECIMEN

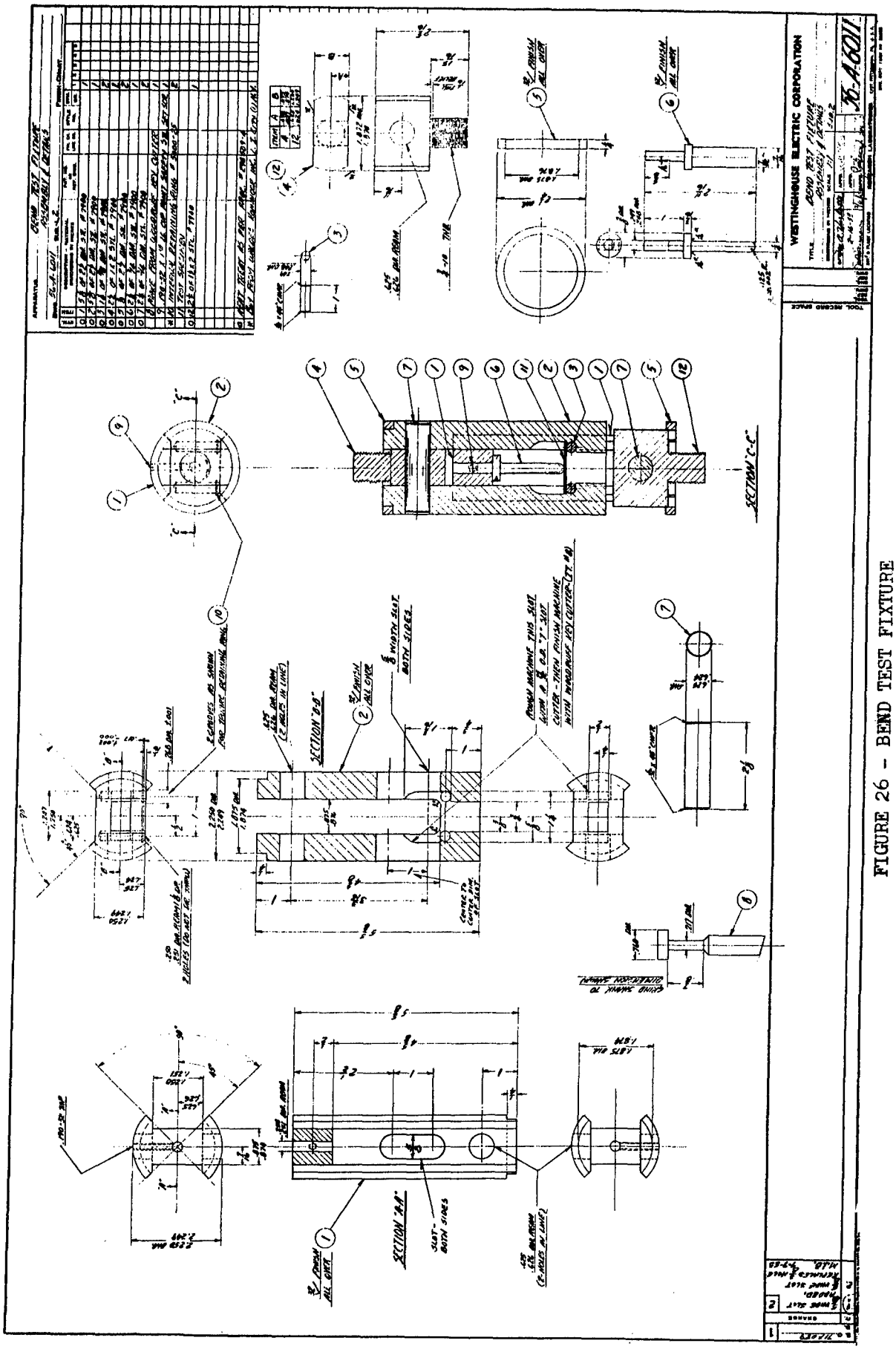


FIGURE 26 - BEND TEST FIXTURE

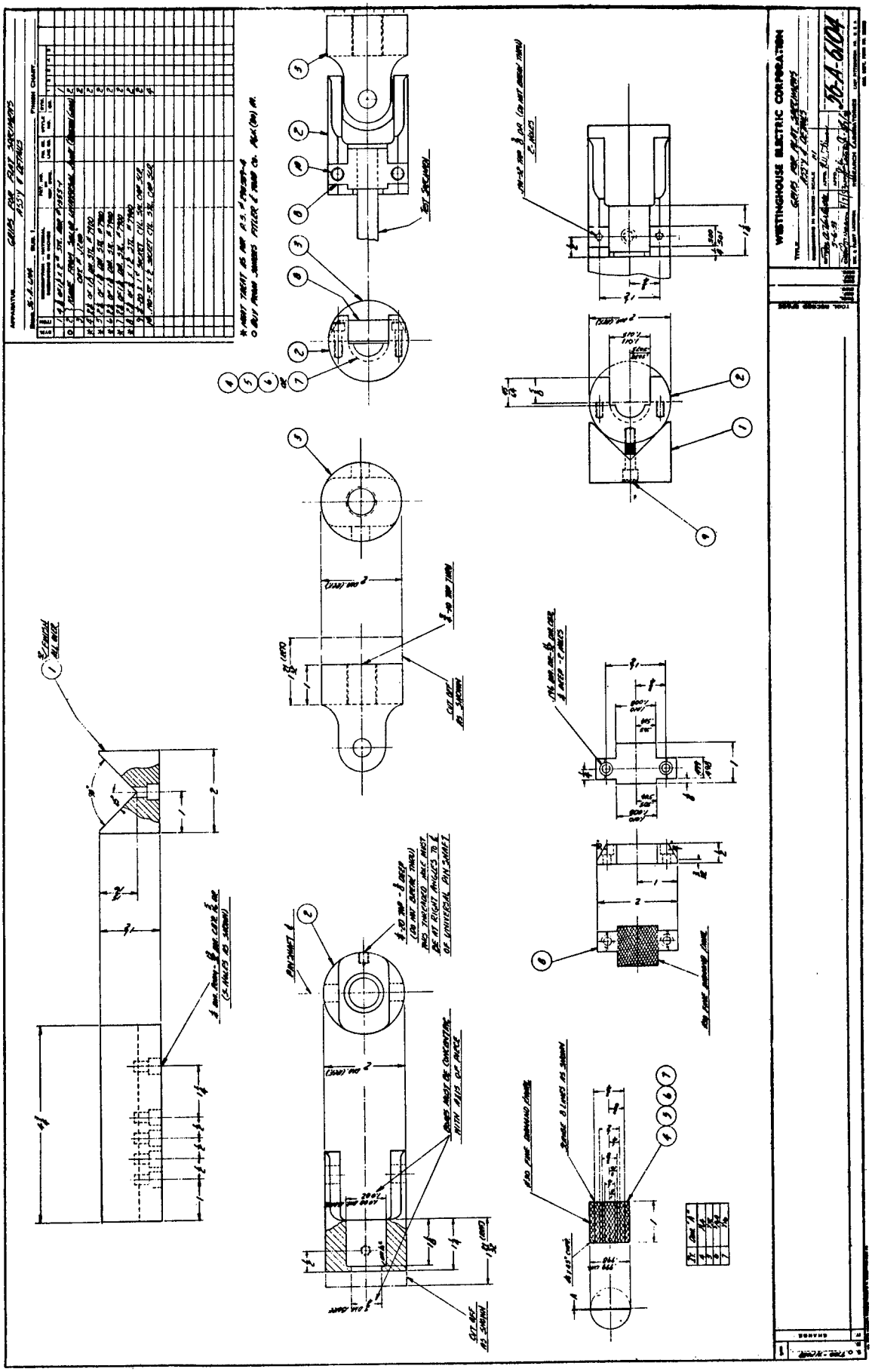


FIGURE 27 - GRIPS FOR FLAT SPECIMENS

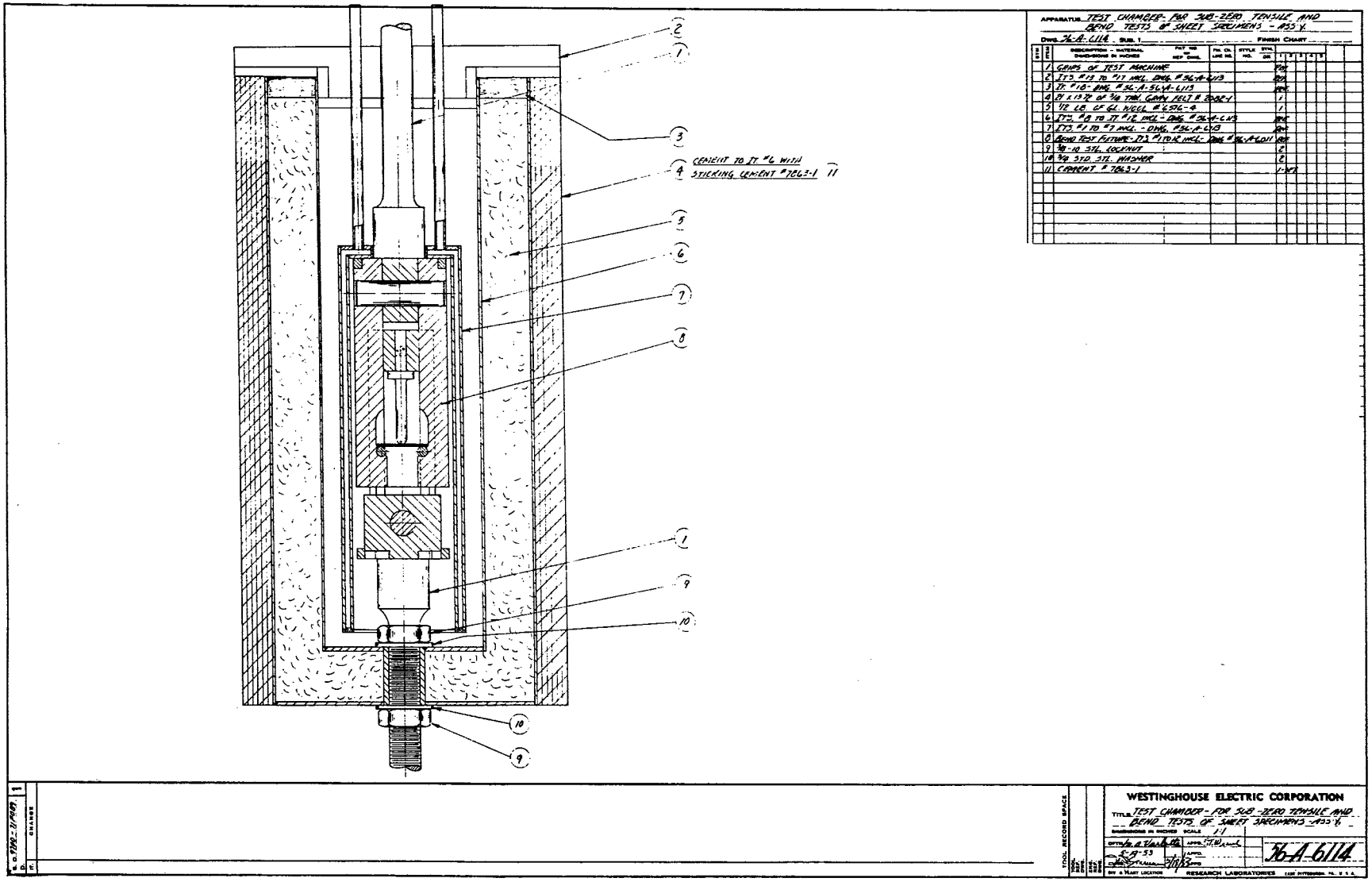


Fig. 28 - TEST CHAMBER-FOR SUB-ZERO TENSILE AND BEND TESTS OF SHEET SPECIMENS-ASS'Y

with the specimen. This thermocouple controls the operation of a solenoid valve which allows liquid N_2 to pass into the double walled intershell. The liquid or gaseous N_2, depending on the temperature desired and the flow rates used, passes through perforations in the inner wall of the double walled chamber. The cold gas then flows down past the sample and bend fixture and works its way out the bottom of the double walled can and ultimately out of the top of the chamber. This device is a modification of a similar unit described by Wessel and Olleman.[4]

In the low temperature and elevated temperature testing chambers only gases are in contact with the specimens. It has been shown by Benedicks[5] that tests performed in liquids which wet the samples may be in error.

IV - WELDING TESTS ON CARBON DEOXIDIZED ARC CAST MATERIAL

Early in the investigation some exploratory tests were made on arc cast and sintered molybdenum sheet. These tests were made in a static He atmosphere. The welding chamber was first pumped down to 10^{-5} mm of Hg and outgassed for 24 hours. Then high purity He was put into the chamber until the pressure was 1 psi. The composition of the gas in the tank used was not available, however, earlier records show that the gas contained 99.98% He, 0.02% N_2 and no O_2. Weld beads were made on samples of sintered molybdenum and carbon deoxidized arc cast material. The weld test pieces were approximately .060 in. x 2 in. x 6 in. The weld bead was made about 4-1/2 in. long and was perpendicular to the final direction of rolling.

All of the weld tests in these investigations were made in the form of beads run on a single plate of molybdenum. No filler metal was added. This procedure was used to avoid external contamination of any sort and in this way the behavior of the weld metal in a single plate could be determined.

This procedure should avoid the effects of several undetermined and uncontrollable variables until the properties of the fused parent metal can be defined. Weld beads were made manually using a 1/8 in. diameter thoriated tungsten electrode. A rectifier type welder was used with the work as the anode (straight polarity). No high frequency arc stabilizer was required. The welding current was 150 amperes and the beads were made without restraining fixtures.

In every instance the arc cast carbon deoxidized material was free from weld bead cracks. However, one deoxidized sample did show a crack which passed through the weld crater. Every weld bead in the non-deoxidized sintered material showed weld bead cracks and gross porosity. Photographs of the welds are shown in Figures 29 and 30. The X-ray photographs of these welds were shown earlier in Figures 1 and 2.

Observation of the weld beads during welding showed cracking and marked gas evolution from the weld puddle in the sintered non-deoxidized material. The puddle in the arc cast deoxidized material was quiet and the bead free from cracks. Repeated passes of the arc over the bead in the sintered material reduced the evolution of gas but porosity was still found at the base of the weld bead at the interface between the fused and unfused material and the beads were cracked. Cracking was observed in the non-deoxidized sintered material during the welding operation. The crack follows the weld puddle at a distance of about one inch. The temperature of the material must have been slightly below the freezing point of molybdenum. The cracks were intergranular which indicates a low melting point grain boundary film as the cause of fracture.

Examination of the weld bead surfaces and transverse microstructures

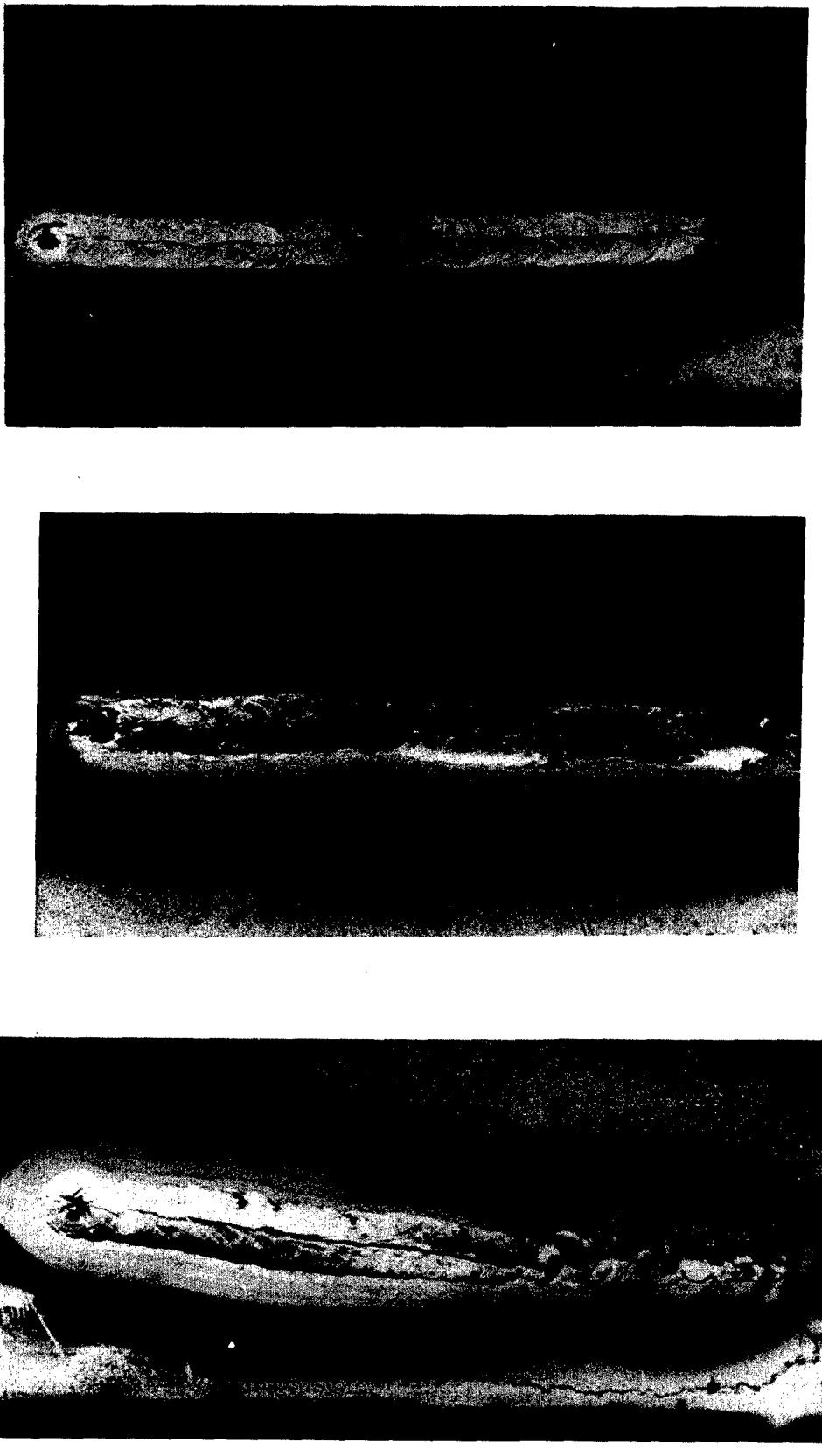


FIG.29 WELD BEADS MADE ON SINTERED NON DEOXIDIZED MO. IN HE. ATMOSPHERE

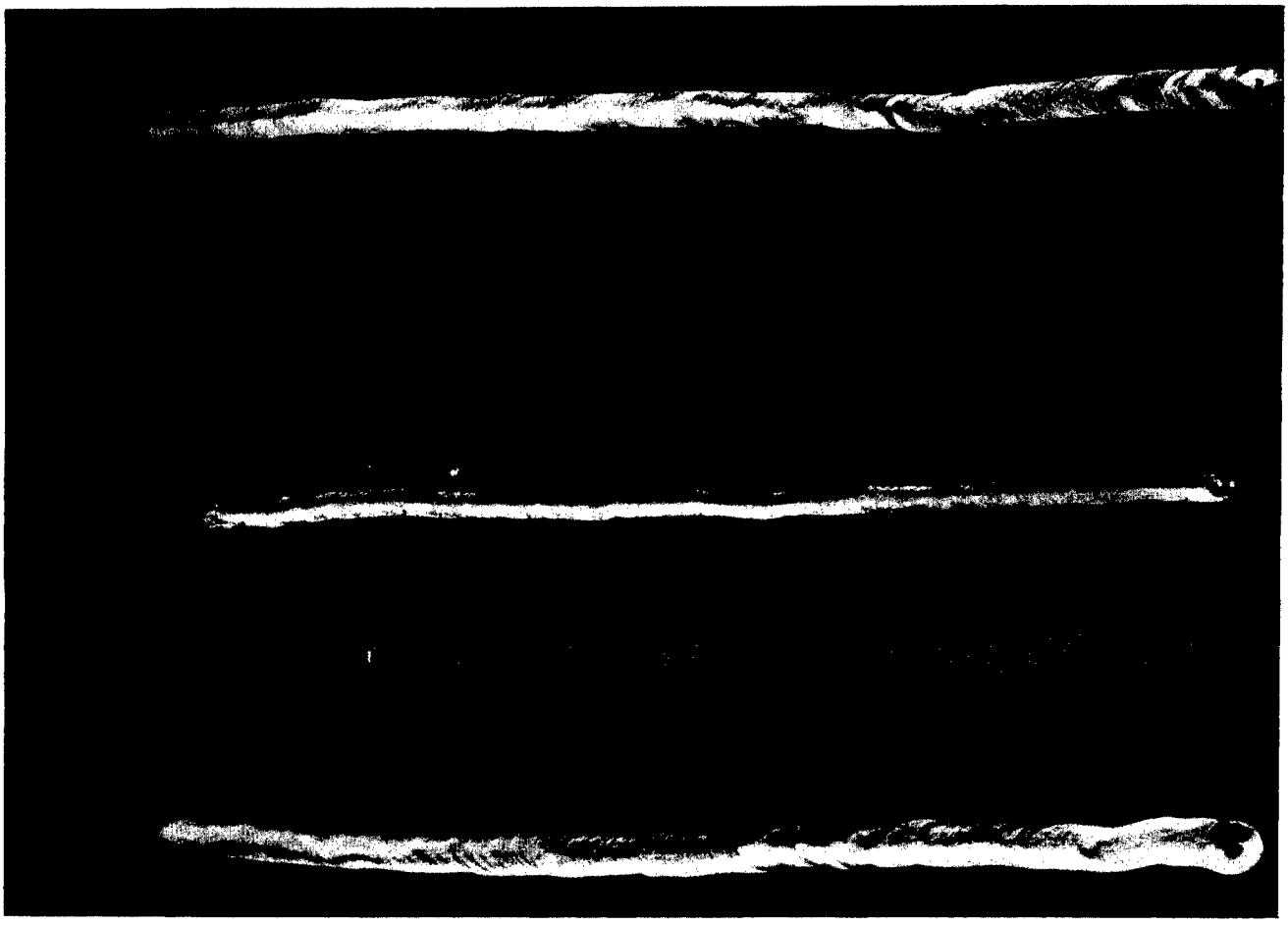
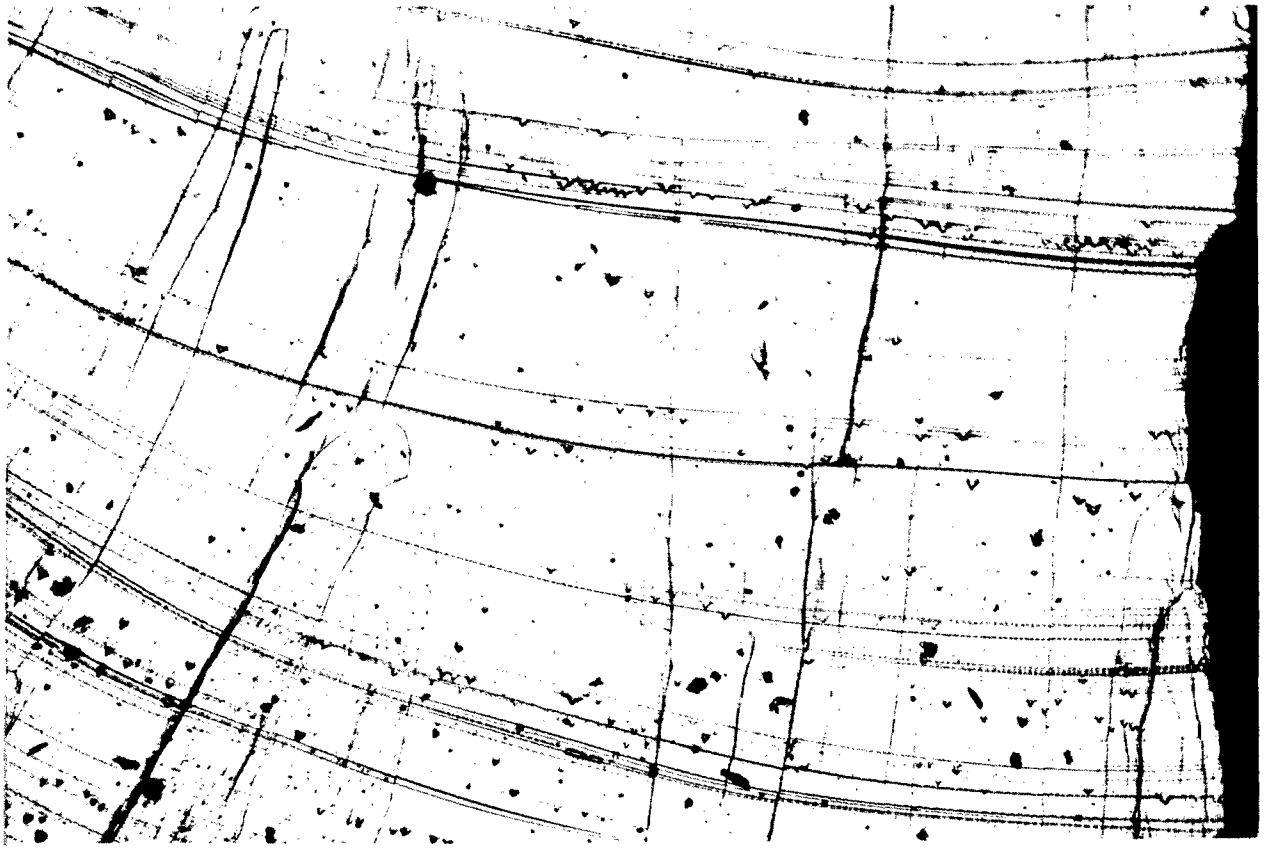


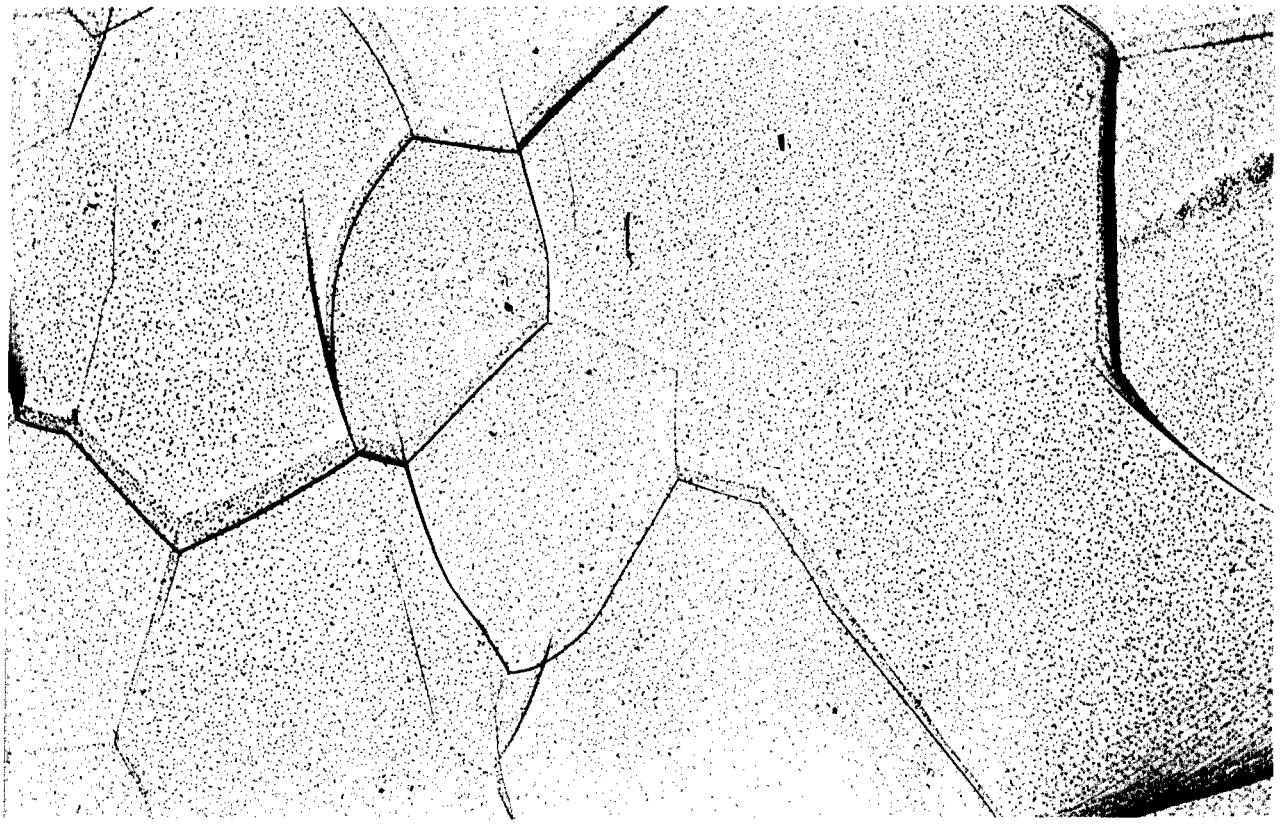
FIG. 30 WELD BEADS ON CARBON DEOXIDIZED MOLYBDENUM MADE IN HE ATMOSPHERE



WELD ARC MOTION

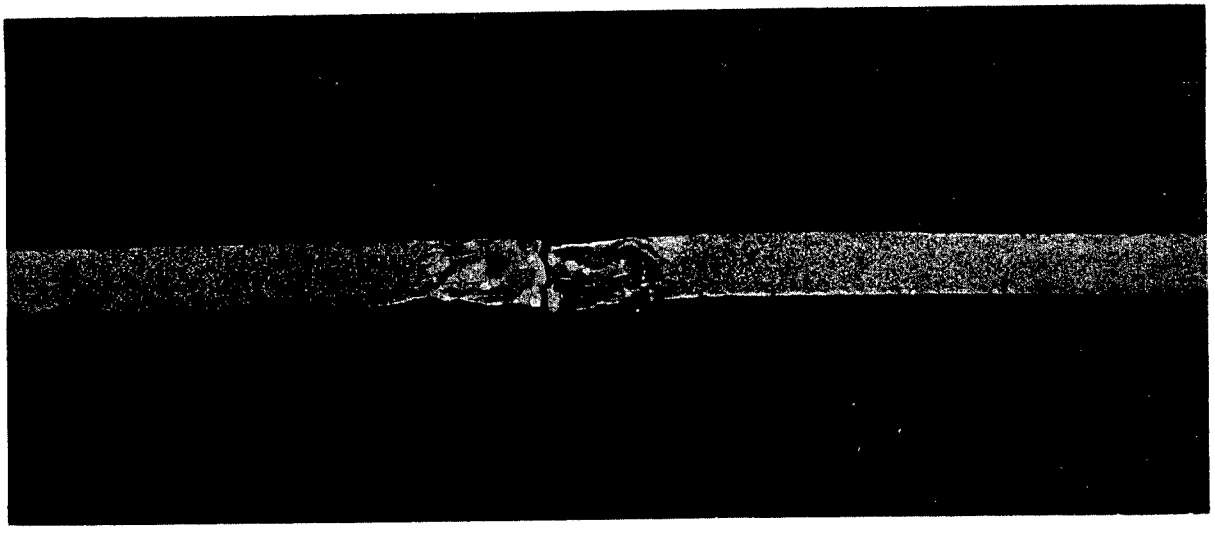
P 53-104-1

FIG.31 WELD BEAD SURFACE UNPOLISHED UNETCHED ON SAMPLE WELDED IN HELIUM ATMOSPHERE SINTERED Mo. NOT DEOXIDIZED 100 X



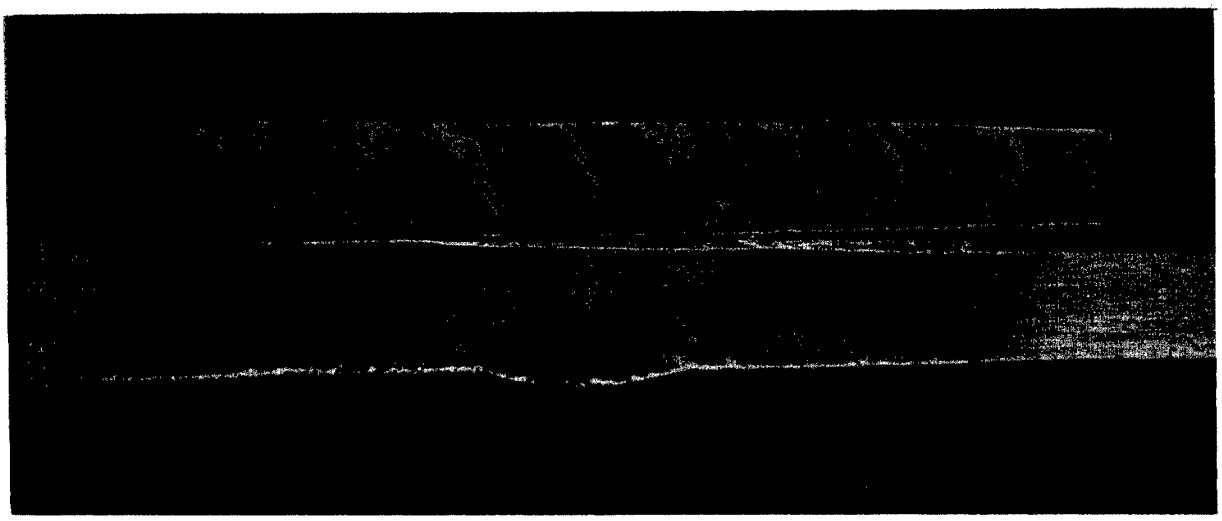
P 53-99-1

FIG. 32 WELD BEAD SURFACE UNPOLISHED UNETCHED ON SAMPLE WELDED IN ARGON ATMOSPHERE ARC CAST Mo. C. DEOXIDIZED 100 X.



P 53-155-3

FIG. 33 WELD IN COMMERCIAL SINTERED MOLYBDENUM MADE IN STATIC He ATMOSPHERE.



P 53-155-2

FIG. 34 WELD IN ARC CAST CARBON DEOXIDIZED MOLYBDENUM MADE IN STATIC He ATMOSPHERE. 10 X

showed a marked difference between the arc cast C deoxidized material and the non-deoxidized sintered material. Photomicrographs of the unpolished, unetched weld bead surfaces are shown in Figures 31 and 32. Photomicrographs of the cross sections of these beads are shown in Figures 33 and 34. The difference in orientation of the columnar weld grains is at once apparent. The arc cast C deoxidized beads have grains perpendicular to the weld surface, and these grains are equiaxed in the plane of the surface. The sintered non-deoxidized material shows a fanlike alignment of grains with the apex of the fan pointing in the direction of arc travel. The grains themselves are roughly parallel to the plane of the sheet. This fan type structure would be expected in the freezing of a pure material under welding conditions. The orientation of the grains in the arc cast material is not easily explained. When 100% penetration occurs grains have been observed which continue from one surface to the opposite surface of the sheet.

The difference in grain orientation may be caused by thermal conditions during welding and cooling or the nature of the material may have caused this difference in orientation. However, in both cases the molybdenum sheet was supported by a copper backup plate. In any event, the fan structure as produced in the sintered material presents a plane of weakness along the center of the weld bead where cracking is known to occur. Definite correlations between the grain orientation and the deoxidization practice, and welding method has not been established.

Examination of welds in sintered molybdenum made in the static He atmosphere showed a surface film on the parent metal near the weld. An X-ray diffraction examination of this film showed it to be molybdenum oxide. Apparently oxygen liberated from the metal during welding increased the

concentration of oxygen around the weld sufficiently to cause oxidization of the metal. In order to prevent this condition a continuous flow of inert gas around the electrode was used on all welds made later in the investigation. In this way local concentrations of O_2 or other impurities was avoided.

A series of welding tests were conducted to determine the effects of oxygen additions to the welding atmosphere. In these tests the oxygen content of the welding atmosphere was deliberately varied from less than 0.05% in welding grade argon to 0.4%. It was found that bead cracking was so severe at 0.4% oxygen that it was felt unnecessary to investigate higher concentrations.

Weld beads were made on the arc cast carbon deoxidized material described earlier in the section on materials. The test samples were blasted with No. 60 Alundum to remove rolling scale and cut to samples .062 in. x 2 in. x 6 in. with the short dimension parallel to the direction of rolling. The beads were made using a 3/16 in. tungsten electrode and a 3/16 in. arc length. The travel speed varied over a range from 7 in./min to 5 in./min. Welding current was held as close to 175 amperes as possible. The travel speed and welding current are interdependent and were established arbitrarily on the basis of 90-100% bead penetration into the molybdenum sheet. The arc voltage changed slightly with the increases in the oxygen content of the atmosphere but was generally about 13 volts. The weld plate was used as the anode (straight polarity). The weld piece was not restrained except by the weight of a hold down block (4 lbs.) used to prevent motion of the plate from vibration or other external motion.

All weld beads were made in the welding chamber described in the section on equipment and shown in Figures 19 and 20. The complete welding procedure data are given in Table IV and the photographs and X-ray pictures of all welds are shown in Figures 35-47.

TABLE IV

WELDING DATA

ARGON OXYGEN TEST WELDS

Test	Atmosphere A	Atmosphere O_2	Atmosphere N_2 and CO	Welding Current Amps.	Welding Voltage	Travel Speed In/min.	Remarks
G-1	99.95	-	-	160	13	-	Insufficient melting.
G-1a	99.95	-	-	180	13	6.2	Repeat bead on top of G1. Distortion. Sample rejected.
G-2	Sample bottle leaked. Input gas welding grade argon.			175	13	6.9	Weld bead bright. Sample clean and free from oxide.
G-3	99.95	.05	Nil	178	13.5	6.9	Welding voltage on before stabilizer. Weld bright and clean.
G-4	Sample bottle leaked air.			180	13	6.5	Weld bright, clean. No oxide on sample or bead.
G-5	99.95	.05	Nil	185	13	7.1	Sample bright and clean, no oxide.
G-6	99.4	0.4	0.2	170	15	6.9	Sample cracked down center of bead.
G-7	99.6	0.3	0.1	180	14	7.1	Weld cracked at ends of bead.
G-8	99.7	0.2	0.1	175	13.5	6.2	Weld cracked down center of bead.
G-9	99.7	0.2	0.1	175	14	6.9	Small crack at start of bead.
G-10	-	-	-	-	-	-	Speed too high, insufficient melting. Sample rejected.
AG-11	99.9	Nil	0.1	185	12	5.6	Sample clean, bright, free from cracks. Sample annealed before welding 1200°C one-half hour.
G-12	99.85	0.02	0.13	180	12.7	5.12	Arc flare at start of weld. Weld not cracked.

TABLE IV
(CONTINUED)

WELDING DATA

ARGON OXYGEN TEST WELDS

Test	Atmosphere A	Atmosphere O_2	Atmosphere N_2 and CO	Welding Current Amps.	Welding Voltage	Travel Speed In/min.	Remarks
G-13	99.9	.05	.05	180	13	5.05	Crack across crater occurred after sample had been removed and was cold.
G-14	99.97	Nil	.03	180	13	5.57	Weld not cracked.
G-15	Welded in air. Conventional torch and shielding.			180	-	5.41	Argon flow 5/liters/min. 10.6 cu.ft./hr.

FIG. 35 SAMPLE G2 ATMOS.-ARGON 99.95 , OXYGEN< 0.05%

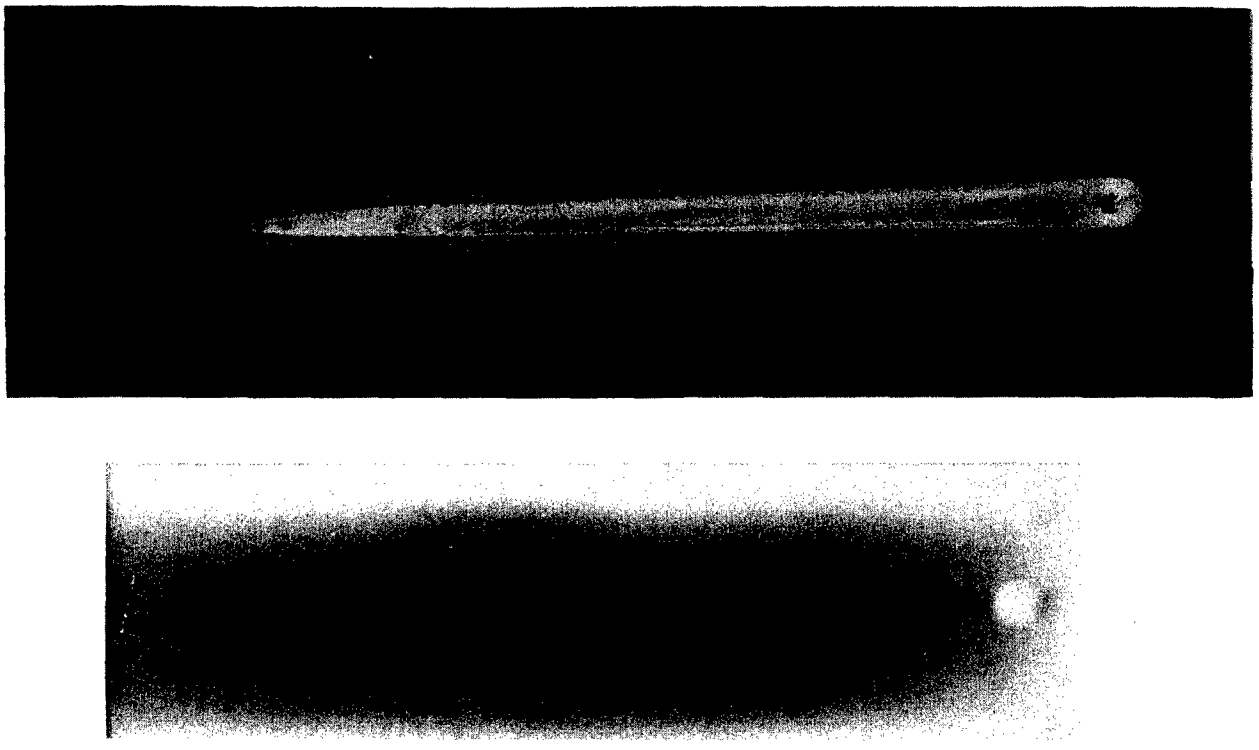


FIG. 36 SAMPLE G3 ATMOS.-ARGON 99.95%, OXYGEN< 0.05%

FIG. 37 SAMPLE G4 ATMOS.-ARGON 99.95%, OXYGEN< 0.05%

FIG.38 SAMPLE G5 ATMOS.-ARGON 99.95%, OXYGEN< 0.05%

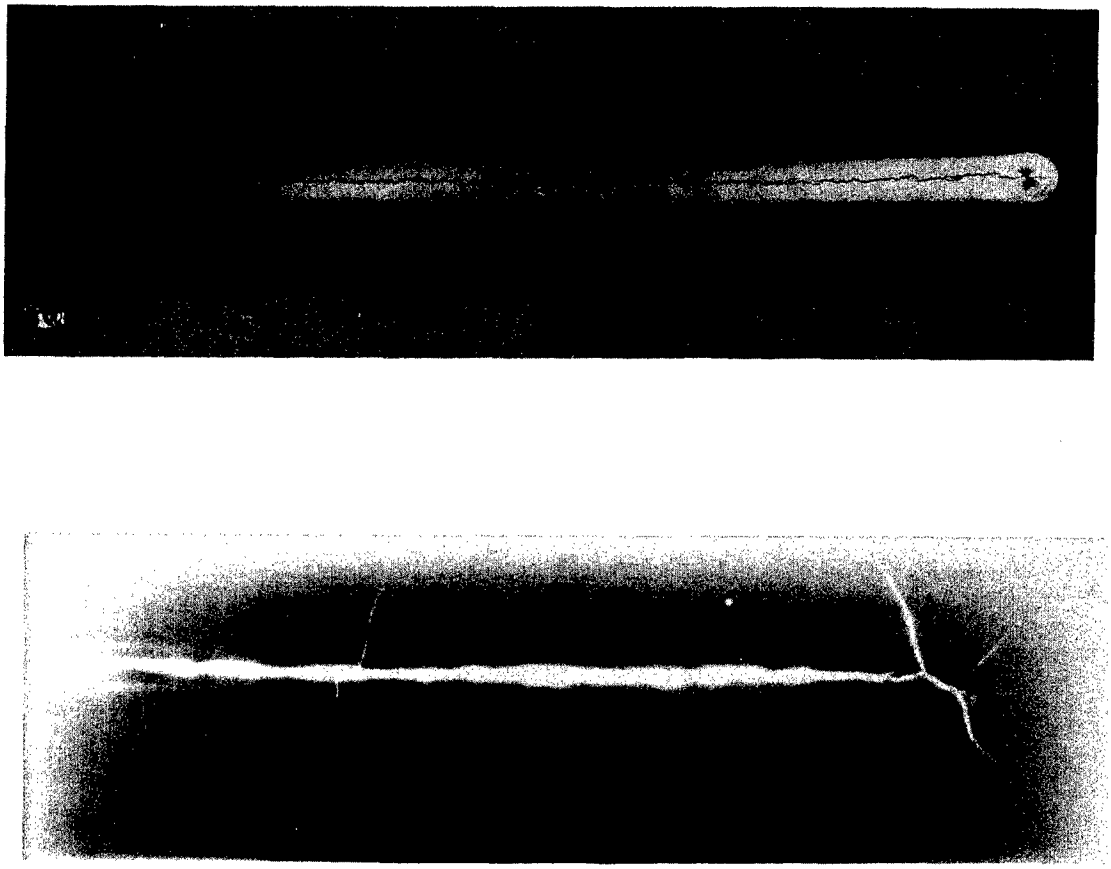


FIG. 39 SAMPLE G6 ATMOS.-ARGON 99.4%, OXYGEN 0.4%, NITROGEN OR CO 0.2%

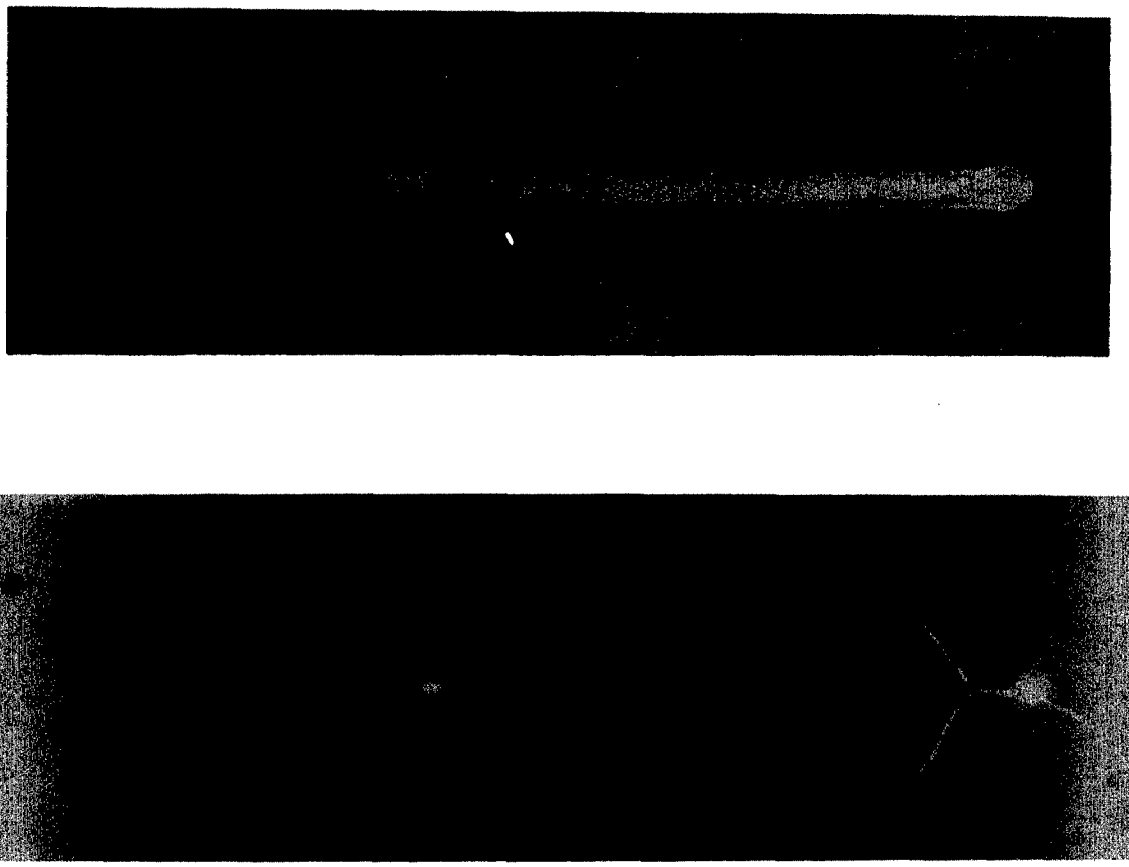


FIG. 40 SAMPLE G7 ATMOS.-ARGON 99.6%, OXYGEN 0.3%, NITROGEN OR CO 0.1%

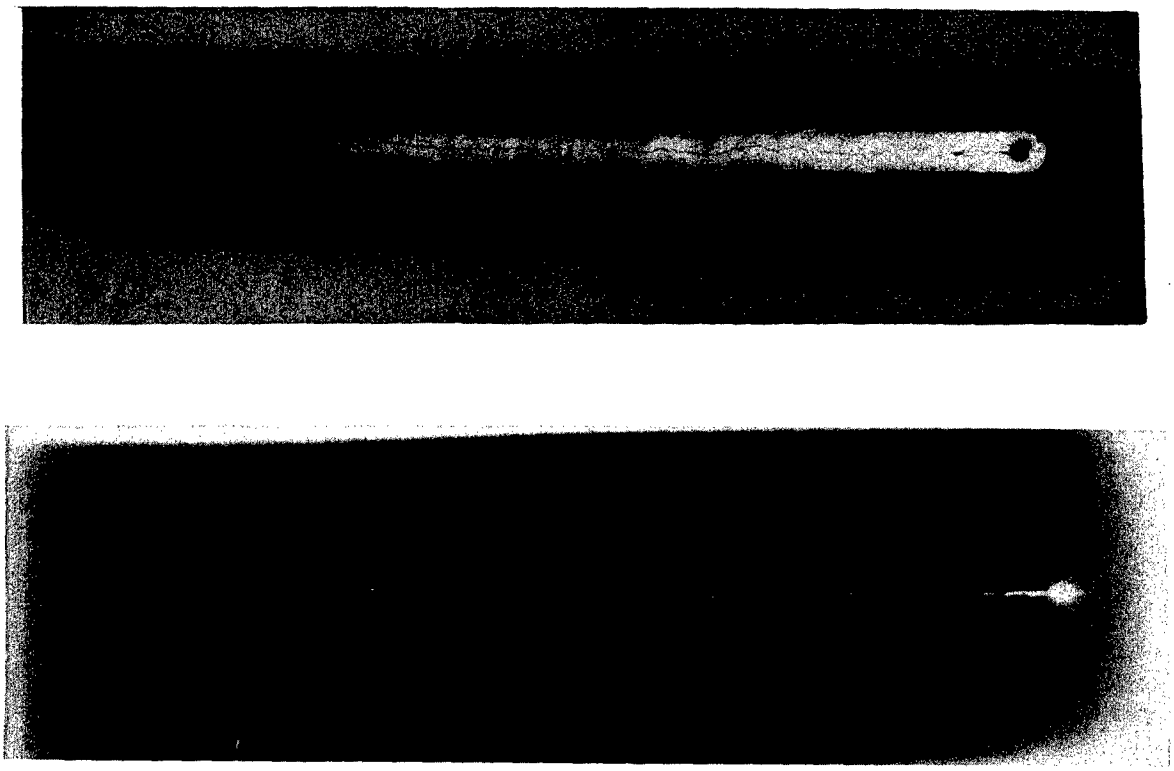


FIG. 41 SAMPLE G8 ATMOS.-ARGON 99.7%, OXYGEN 0.2%, NITROGEN OR CO 0.1%

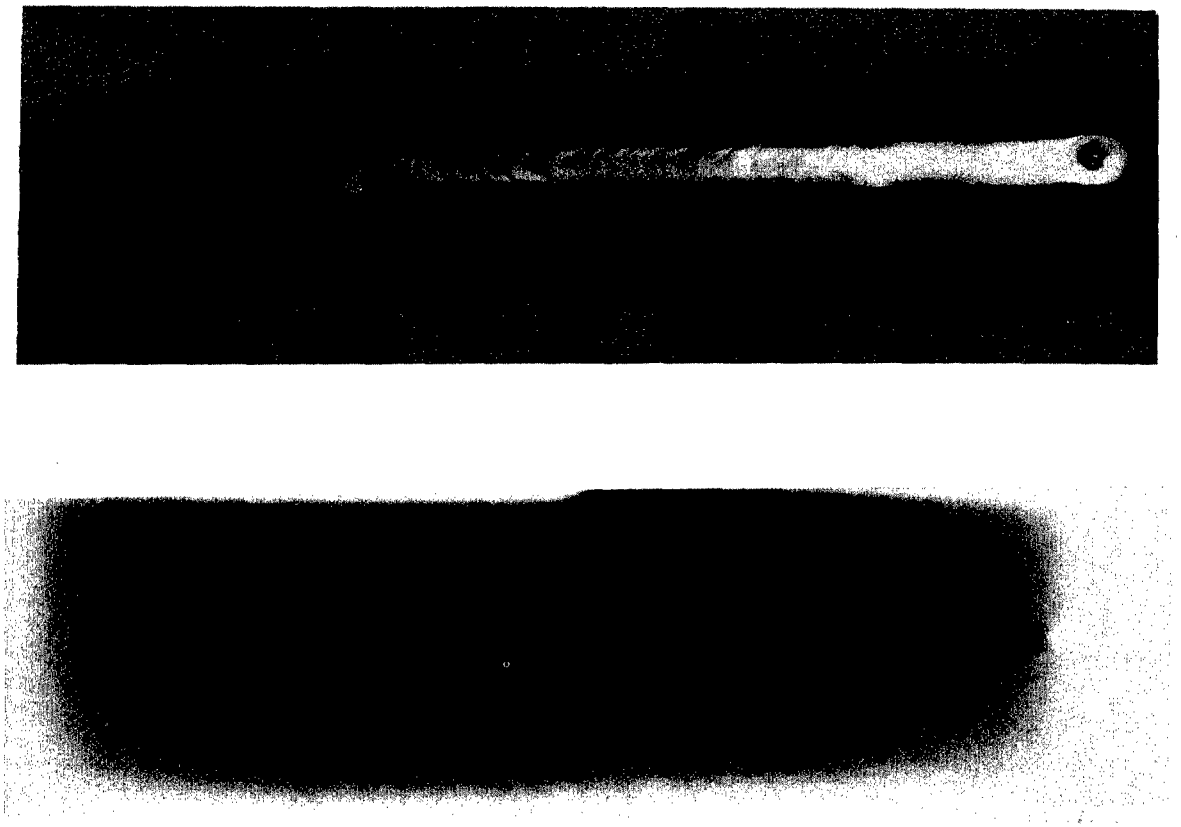


FIG. 42 SAMPLE G9 ATMOS.-ARGON 99.7%, OXYGEN 0.2%, NITROGEN OR CO 0.1%

FIG. 43 SAMPLE AG11 ANNEALED 1/2 HR. 1200°C BEFORE WELDING. ATMOS-ARGON 99.95%.

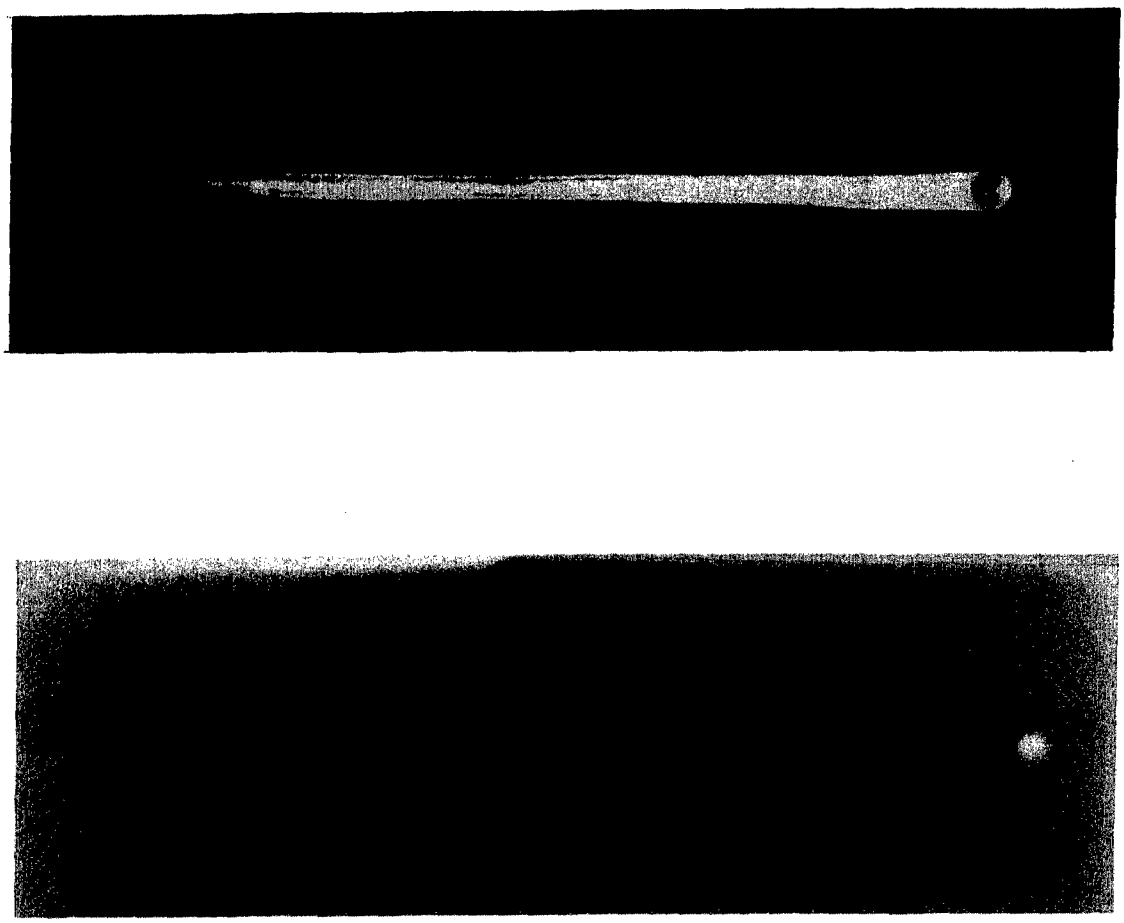


FIG. 44 SAMPLE G12 ATMOS.-ARGON 99.85%, OXYGEN .02%, NITROGEN OR CO .13%

FIG. 45 SAMPLE G13 ATMOS.-ARGON 99.9%, OXYGEN .05%, NITROGEN OR CO .05%

FIG. 46 SAMPLE G14 ATMOS.-ARGON 99.97%, OXYGEN NIL, NITROGEN OR CO .03%

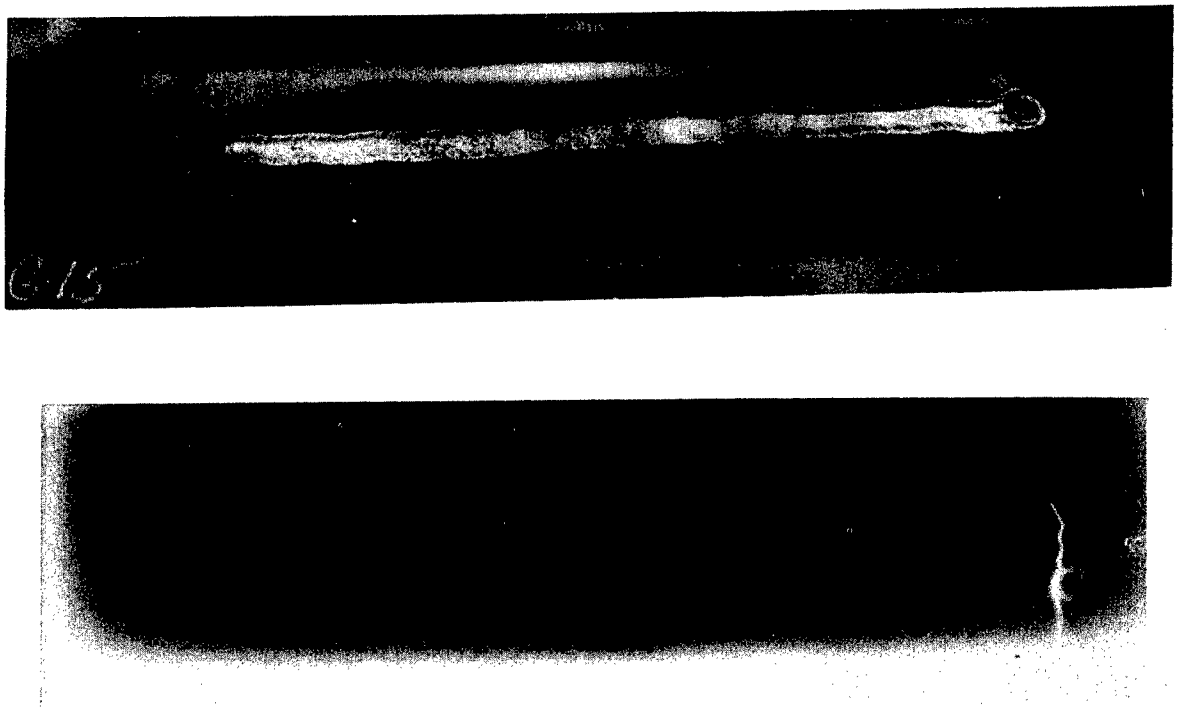


FIG. 47 SAMPLE G15 WELDED WITH CONVENTIONAL ARGON SHIELDED TORCH. GAS FLOW 10.6 CU FT / HR.

Four weld beads were made using "as rolled" carbon deoxidized molybdenum sheet in an atmosphere of welding grade argon 99.95% with less than 0.05% O_2. These four samples, G2, G3, G4, G5, are shown in Figures 35-38. Examination of these figures shows no evidence of cracks or porosity. The ability to produce four successive beads without defects was taken as an indication that such beads could be reproduced at any time that the same welding conditions are used.

Deliberate O_2 additions were made to the welding atmosphere when samples G6, G7, G8, G9, G12, G13 and G14 were welded. The effects of oxygen in the welding atmosphere is shown very clearly in Figures 41 and 42. The addition of as little as 0.2% oxygen to the atmosphere may be sufficient to produce hot short cracking in the arc cast C deoxidized molybdenum. Apparently there is some scatter in the data since cracking was found in samples G8 and G9, Figures 41 and 42, with 0.2% O_2 added to the atmosphere, while sample G7 was not cracked with 0.3% O_2 added. This discrepancy may be a function of thermal stress effects or other unknown factors. Examination of the two beads made with 0.2% O_2 added to the atmosphere shows definite cracking along the entire length of the bead in the case of sample G8, Figure 41. Sample G9, Figure 42, showed only a short crack near one end. It appears that the cracking threshold for O_2 additions to the welding atmosphere is at or very near 0.2%.

Visual examination of these cracks at magnifications up to 30X shows that the fractures are of a grain boundary nature. Since cracking occurs while the beads are hot and the cracks are intercrystalline in nature, it is apparent that O_2 in the welding atmosphere produced low melting point constituents, probably oxides, in grain boundaries. From the general nature of the cracks and their propagation behind the weld puddle, it is apparent

that the grain boundary layer had not yet solidified, or that they were very weak at the temperatures where drastic thermal stresses occur during cooling. These data show that weld beads made in the presence of 0.2% O_2 or more are definitely hot short.

The absence of porosity in the welds made with O_2 additions to the atmosphere is believed to be due to the formation of oxides by absorption at the bead surface during fusion rather than evolution from the weld metal. However, it is also possible that the porosity observed in sintered molybdenum weld beads may be due to gases other than oxygen.

Two types of cracking appear in samples welded in atmospheres containing additions of oxygen. Cracks occur in the center of the bead and perpendicular to the bead. The latter may extend for a considerable distance into the unfused material. Strictly speaking, cracks through the weld crater at the end of the bead are not of the cross crack type since they originate in an area of reduced section at the crater and may or may not be perpendicular to the weld bead. Cross cracks which do not occur in the weld crater may be found in samples having no center bead crack. However, this type of cracking frequently occurs in conjunction with center bead cracking. Sample G7, Figure 40, shows an example of cross bead cracking in the absence of a center bead crack. It should be noted that for this sample the oxygen in the atmosphere was sufficiently high to have produced a center bead crack, 0.3%, as in sample G8, Figure 41. Apparently both cross bead and center bead cracks are a function of the oxygen content of the atmosphere surrounding the weld in arc cast C deoxidized material.

Crater cracking is probably also a function of the oxygen content of the atmosphere. Of the samples welded cracks in the craters have occurred only in the cases of deliberate oxygen additions. Samples G6, G7, G13,

Figures 39, 40 and 45, all show crater cracking. Samples G2, G3, G4, G5, AG11, G12, and G14, Figures 35-38, 43, 44 and 46, show no crater cracks. The first group were all made with deliberate oxygen additions. The second group with the exception of sample G12, Figure 44, were made in welding grade argon without O_2 additions. In the case of samples G12 and G13, Figures 44 and 45, there is some question as to the oxygen content of the atmosphere. The analysis for sample G12 shows O_2 0.02% and sample G13 shows O_2 0.05%. With the present mass spectrometric equipment it is difficult to obtain accurate analyses for gases present in amounts less than 0.05%. In some instances the noise level on the mass spectrometer obscures the readings below 0.05%. However, in the cases of samples G12 and G13, known additions of oxygen were made to the welding grade argon before it entered the welding chamber. The intended atmosphere compositions in these two cases were 0.01% and 0.08%, respectively, as compared to the analytical data showing 0.02 and 0.05%. There is a good probability that the spectrometer readings obtained for these two samples are reasonably accurate.

One weld bead was made on arc cast C deoxidized molybdenum sheet using a conventional argon arc shielded torch in air. The argon flow rate was 10.6 cu ft/hr. The welding current was 180 amperes, and the travel speed approximately 4.5 in./min. The photograph and X-ray picture of this weld is shown in Figure 47. The cross crack at the crater indicates oxygen pickup. The sample was heavily oxidized, apparently after the shield moved away from the frozen metal. Oxidization of the surface apparently occurred only after the weld was incapable of absorbing sufficient oxygen to produce center bead cracking.

The oxygen contents of the weld beads have not been investigated.

However, future work along these lines is planned when analytical techniques are sufficiently improved to reveal significant differences.

The discussion of weld bead cracking should include some mention of the stresses involved in a weld test plate of the type used in these investigations. A study of residual stresses in welds using a photoelastic model has shown that weld stresses in samples of the type used had residual stress concentrations at the center and ends of the beads.[7] M. Mark showed that there is a marked stress concentration at the center of the weld bead in the area where center bead cracking occurs. He also showed stress concentrations in the area of the crater which would tend to produce crater cracking. These data indicate that the type of weld samples used in the molybdenum joining program constitutes a relatively rigid physical test by their very nature. This does not imply that further tests are unnecessary but indicates that if welds are not cracked after welding they have some degree of ductility.

The weld bead samples were welded in the "as rolled" condition. Residual stresses were undoubtedly present, although the test plates did not distort to an observable degree. The material recrystallized during welding at a distance of about 3/4 in. on each side of the bead. This recrystallization indicates temperatures above 1300°C at the maximum distance from the weld bead. Such temperatures should relieve rolling stresses while imposing new and greater thermal stresses. Residual rolling stresses were known to exist in the unwelded sheet because distortion was encountered when "as rolled" sheets were surface ground under good cooling conditions. The distortion indicated that the surfaces of the specimens were in tension. Similar grinding operations on samples recrystallized for one hour at 1200°C showed

no distortion.

Bend tests have been made on 2 welds made in arc cast C deoxidized molybdenum. These tests covered a range of temperatures from -130°F to +500°F (-90°C to +260°C). A similar test was made on recrystallized unwelded sheet. Weld samples G5 and G7, Figures 38 and 42, were used to make bend specimens of the type shown in Figure 23. These specimens were bent at a deflection rate of 6 in./hr, with the exception of weld samples G5-1 and G5-2 which were deflected at a rate of 5 in./hr. The results of these bend tests are given in Tables V, VI and VII. The results are also shown in graphic form in Figures 48 and 49. The effect of oxygen additions to the welding atmosphere upon the ductile to brittle transition temperature range becomes obvious from these data. Increases in the oxygen content in the welding atmosphere seem for these limited data to widen the transition temperature range as compared to that of the unwelded recrystallized material without an appreciable change in the temperature of completely brittle failure. It should be noted that even the small amounts of O_2 present in the welding grade argon may have caused a loss in weld ductility.

Bend tests on sample G9 were limited to a maximum testing temperature of 500°F (260°C) by the testing fixture. The ductile to brittle transition temperature for sample G9 would probably have fallen in the neighborhood of 600-700°F (315-371°C). Such transition temperatures become academic in an investigation of this type in which the objective is to lower the transition temperature range. However, the marked increase in the transition temperature range by oxygen additions to the atmosphere is of considerable importance.

Bend test data are subject to certain limitations. With the fixture and specimens used the maximum deflection obtainable was 0.4 in., beyond

TABLE V

BEND TEST DATA FOR MOLYBDENUM

WELD G-5

Spec. No.	Test Temp. °C	Deflection Rate	Thickness Ins.	Width Ins.	Prop. Limit	Maximum Load	Fracture Load	Deflection at Fracture	Type Fracture*
G-5-1	30	5"/hr.	.0635	.2465	55.7	82.8	82.8	0.0985	TC IC
G-5-2	-10	5"/hr.	.0635	.2540	73.1	102.6	102.6	0.0665	IC
G-5-3	-50	6"/hr.	.0635	.2480	90.0	101.0	101.0	0.0054	TC
G-5-4	-90	6"/hr.	.0635	.2445	-	88.0	88.0	0	TC
G-5-5	+70	6"/hr	.0635	.2530	34.0	73.0	-	-	No Fracture
G-5-6	+50	6"/hr.	.0635	.2490	45.0	71.5	73.5	0.0762	Cracked-No Fracture
G-5-7	+60	6"/hr.	.0635	.2555	42.0	75.0	75.0	0.1290	IC
G-5-8	+77	6"/hr.	.0636	.2540	44.0	71.5	71.0	0.2191	TC IC

*TC - Transcrystalline Fracture

IC - Intercrystalline Fracture

TABLE VI

BEND TEST DATA FOR ANNEALED ARC CAST MOLYBDENUM SHEET

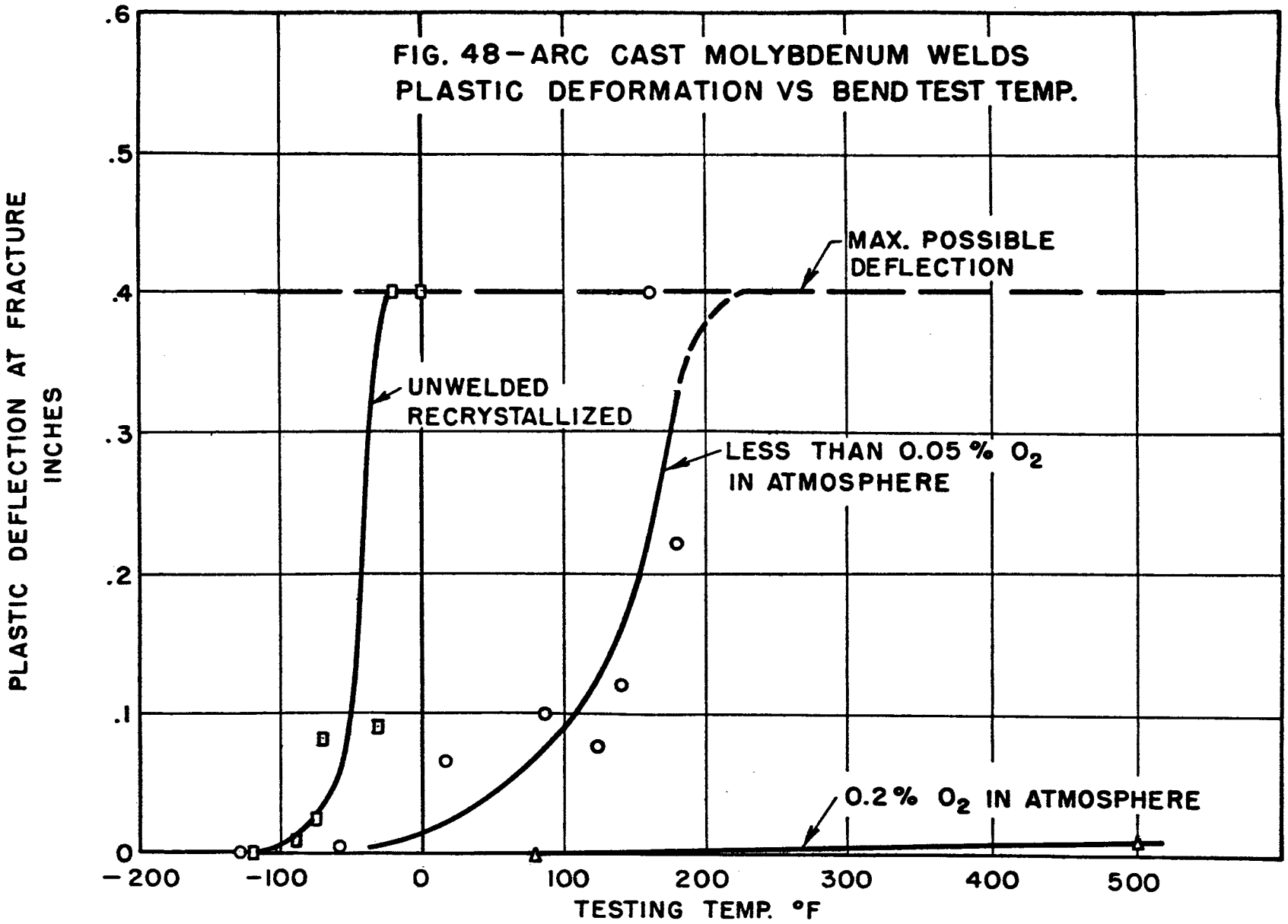
Spec. No.	Test Temp. °F	Deflection Rate	Thickness	Width	Load at Proportional Limit	Load at Fracture	Deflection at Fracture
5143-1-1	-120°F	6"/hr.	.254	.2595	-	111 lbs.	-
5143-1-2	- 20°F	6"/hr.	.247	.2650	93	114*	No Fracture
5143-1-3	- 70°F	6"/hr.	.2525	.260	95	125	.083
5143-1-4	- 90°F	6"/hr.	.250	.260	103	114	.009
5143-1-5	- 50°F	6"/hr.	.250	.259	93	107	.020
5143-1-6	- 30°F	6"/hr.	.250	.260	94	115	.092
5143-1-7	- 0	6"/hr.	.2525	.2595	80	107*	No Fracture

*Maximum Load.

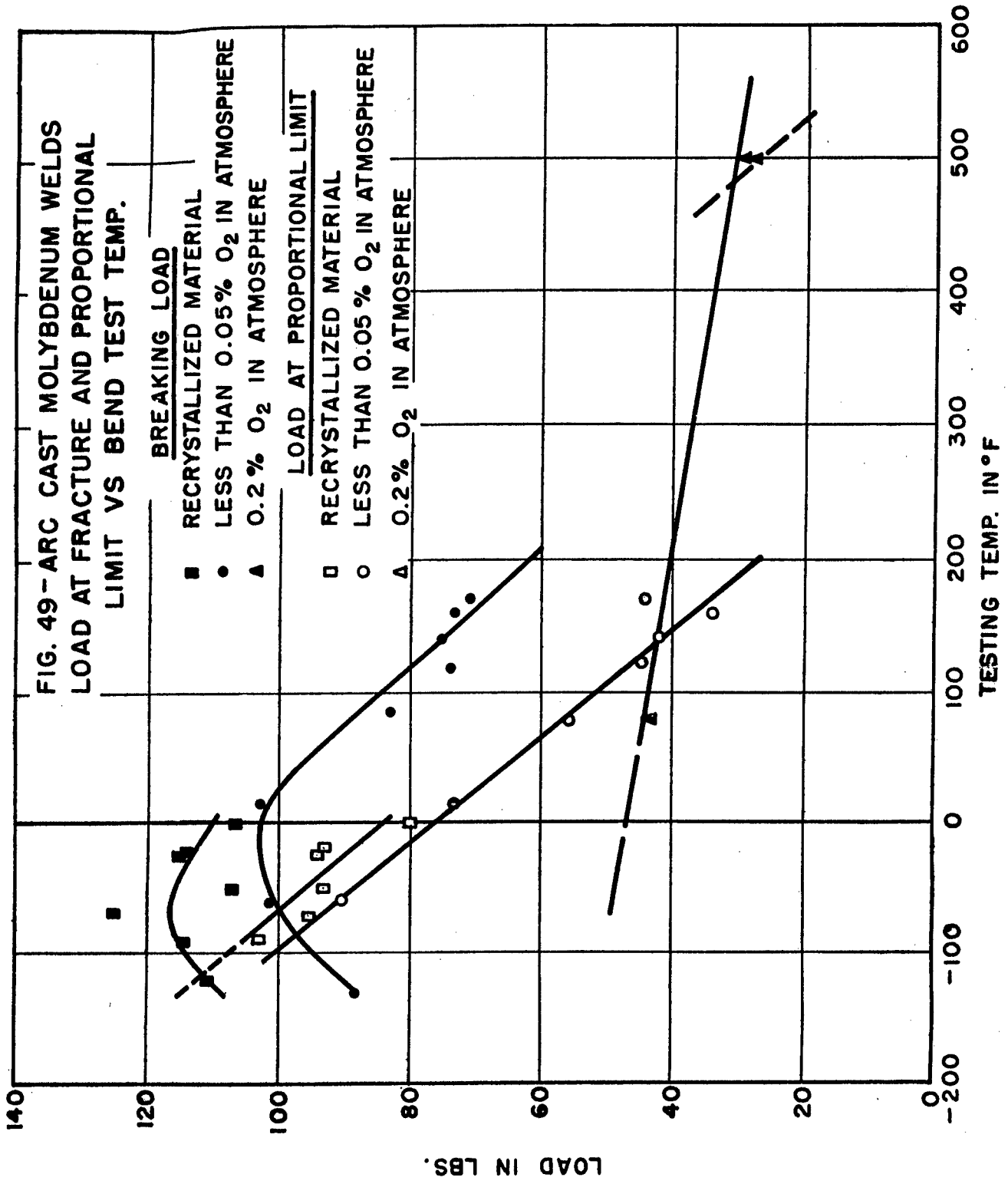
TABLE VII

BEND TEST DATA FOR MOLYBDENUM WELDED IN ATMOSPHERE CONTAINING 0.2% O_2

Spec. No.	Test Temp. °F	Deflection Rate	Thickness	Width	Load at Proportional Limit	Load at Fracture	Deflection at Fracture
G-9-1	80°F	6"/hr.	-	-	-	43	-
G-9-2	500°F	6"/hr.	-	-	12	30	.013



CURVE 383929



CURVE 383930

this point the sample is pushed through the fixture. Thus, the reported data must be considered with the limitations of the fixture in mind. Also, after the specimen has locally deformed at an area opposite the end of the ram, further deformation occurs away from the original location of strain. Thus in this particular case the weld area will be deformed more or less uniformly, but the maximum deformation at any point in this area will not be excessive. The maximum total bend angle attainable is 126°. This angle of bend on the samples used produces a radius of curvature of about 0.18 in. If it is assumed that the neutral axis does not shift toward the compression side, then the distance to the neutral axis is thickness/2 or 0.0318 in. The elongation then would be of 17.6% from the equation

$$\frac{\varepsilon}{\frac{t}{2}} = \frac{1}{\rho}$$ where ε is strain, t = thickness of the sample, and ρ is the radius of curvature of the neutral axis.

The rate of deformation also places further limitations on the bend test. If, as in these tests, a constant rate of ram motion is used, the surface area opposite the ram will have a strain rate which may rise rapidly to a maximum and then fall off to a low value as shown in Figure 50. The shape of this curve is only intended to be qualitative in nature. If the strain rate at the center of the bend specimen is approximately as shown in Figure 50, then it can be seen that the bend test will be extremely sensitive to brittle behavior. On the other hand, when the ductility exceeds the relatively small amount required by the radius of curvature of the ram and the friction of the system, the bend test would show what appears to be completely ductile behavior.

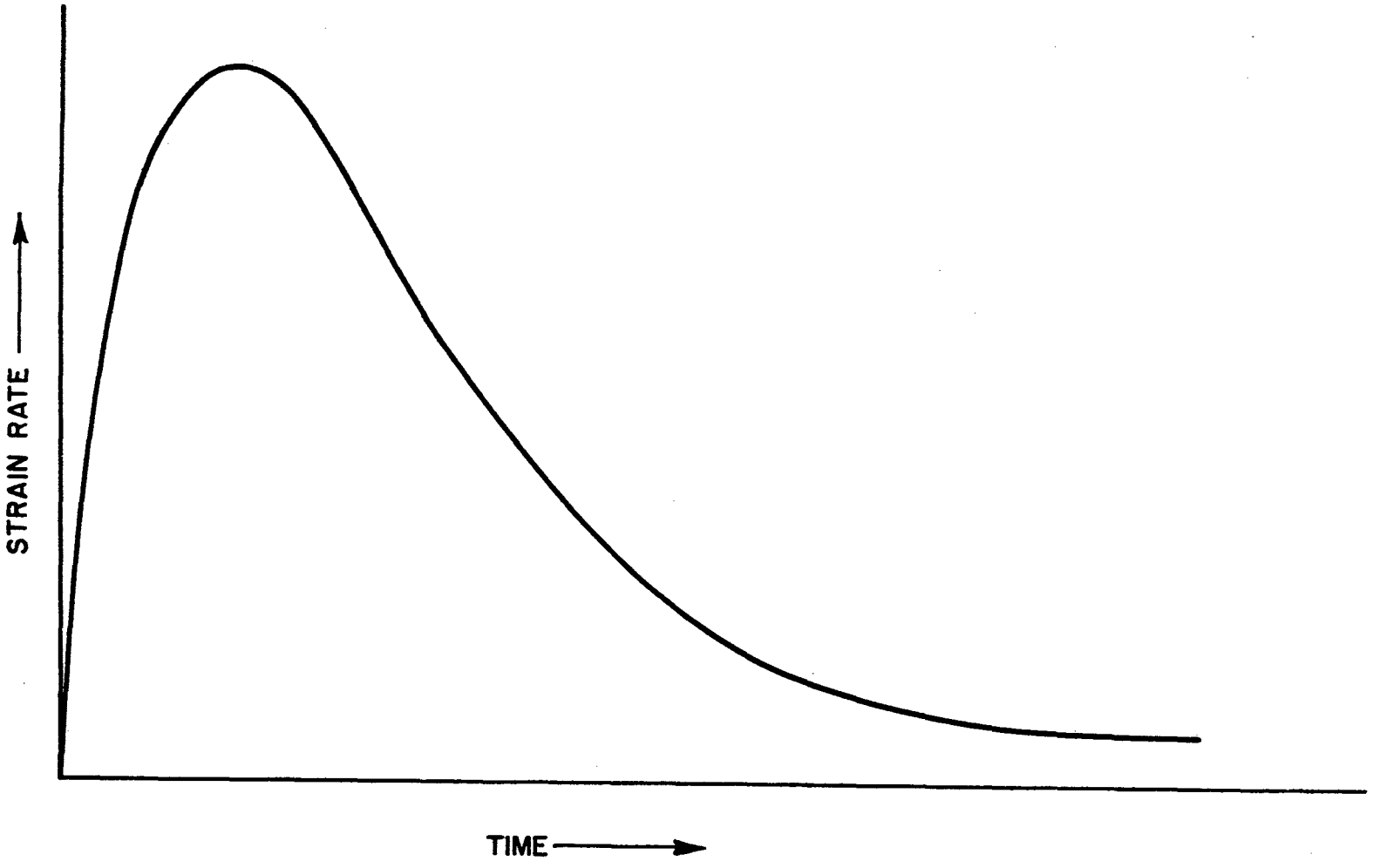


FIG. 50—PROBABLE RELATIONSHIP BETWEEN STRAIN RATE AND TIME FOR BEND TESTS.

When the limitations of the bend test are carefully considered, it is apparent that this type of test will show relative improvement in ductility when the materials are in the low ductility zone. Thus this test shows the effect of changes from brittle to ductile behavior over a smaller temperature range than does the tensile test. In the bend test as the ductility nears or exceeds the amount necessary to permit deformation around the bending ram, the material will appear to be ductile. Actually, the ductility may be relatively low as compared to tensile tests. However, ductility which will permit bending around a radius equal to twice the thickness of the sheet should be adequate to avoid moderate impact shock failures at the same temperature.

From the data shown in Figures 48 and 49 and the considerations just mentioned in connection with the bend test, it appears that the ductile to brittle transition temperature range in welds made in deoxidized molybdenum with welding grade argon, 99.95% A, with less than 0.05% O_2, will give reasonable engineering ductility at temperatures above 200°F and show a ductile to brittle transition range in bending around a radius twice its thickness at temperatures between +200°F and -130°F where it becomes completely brittle. From the curve for less than 0.05% O_2 in Figure 48, the welds will probably be capable of some deflection or slight shock at temperatures from 32°F to 100°F, but may be subject to brittle rupture in this transition range.

V - WELDING TESTS ON DEOXIDIZED SINTERED MOLYBDENUM

Improvement of sintered molybdenum to the extent that it may be welded without cracks and porosity is an important phase of the molybdenum joining program. The preparation of vacuum sintered molybdenum ingots which were

deoxidized with several different minor alloy additions was discussed earlier under Material. Weld beads were made on test pieces prepared from these vacuum sintered deoxidized ingots. The weld beads were made using welding grade argon (99.95% A) in the welding chamber discussed under equipment and shown in Figure 20. Unfortunately, the sintered deoxidized samples were only 1-1/4 in. wide because of sintering limitations. These narrow samples were difficult to position and hold with existing fixtures. Satisfactory welding conditions were, therefore, not always obtained.

The deoxidizers used in ingot preparation appeared to produce different melting requirements in the molybdenum sheet. For this reason it was not possible to make necessary arc current adjustments before the weld was started.

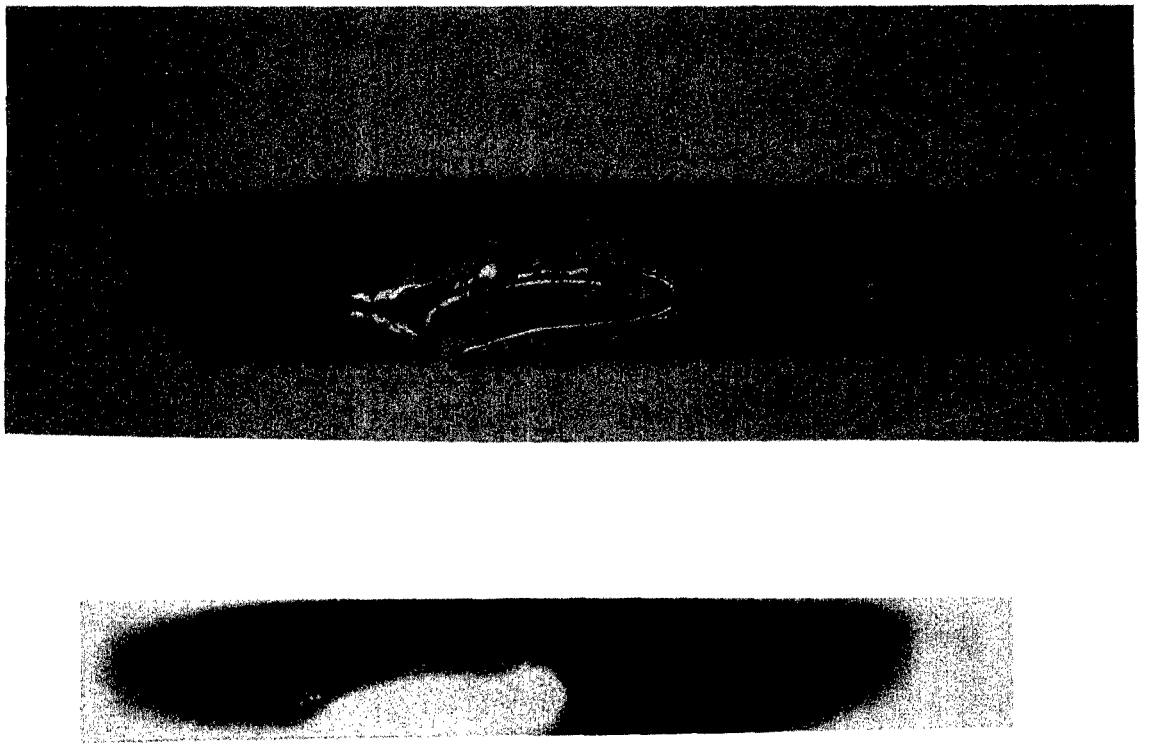
The weld beads made in the sintered deoxidized plates are shown in Figures 51 through 56. Radiographs of these welds are shown beside their photographs. Welding data and sample analyses are given in Table VIII. These welds should not in any sense be considered as having satisfactory quality because of the experimental difficulties encountered. However, they do provide valuable exploratory data. A comparison of the radiographs of the deoxidized weld beads with a weld bead made in commercial sintered molybdenum from earlier tests, Figure 1, shows that the deoxidization practice has reduced the weld porosity and may have reduced the hot short cracking. However, the difference in sample width and resultant reduction in thermal stresses in the deoxidized sheets may account for the absence of hot short cracks. It is probable, however, that deoxidization aided in the elimination of weld cracks.

It is difficult to compare the weld beads on these deoxidized samples because of the large experimental deviation. Welding requirements apparently changed with the deoxidizing element used. However, the pure vacuum sintered

TABLE VIII

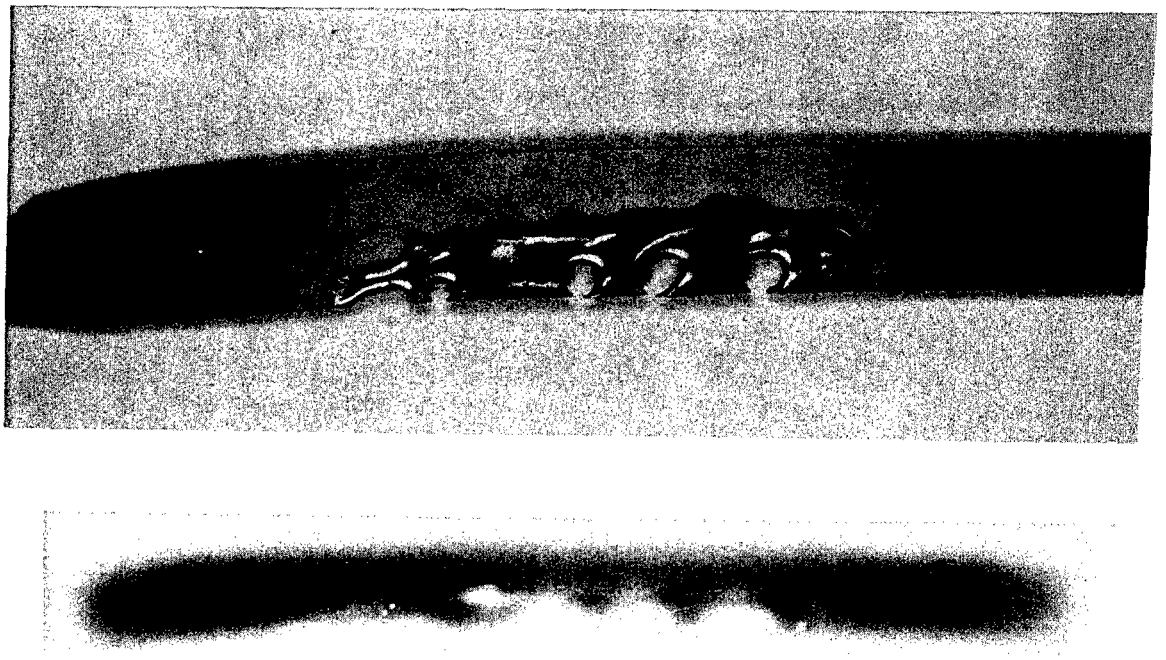
WELDING DATA FOR VACUUM SINTERED MOLYBDENUM

Sample	Sheet Analysis	Welding Current	Travel Speed	Atmosphere Analysis	Comments
WS 146	0.12%C	140-120 Amps.	2.5"/min.	O_2 .01 N_2 or CO 0.1%	Too hot current reduced during welding.
WS 147	0.3%C	160 Amps.	6.6"/min.		Hot, melted through.
WS 148	0.10% Ti	160 Amps.	6.6"/min.	O_2 .01% N_2 or CO .06%	Hot. Partly melted through.
WS 149	0.5% Ti	160 Amps.	6.6"/min.	O_2 .01% N_2 or CO 0.1%	Good smooth bead.
WS 150	0.09% Al	160 Amps.	6.6"/min.	O_2 .01% N_2 or CO .04%	Had to repeat pass because of improper speed adjustment.
WS 151	0.18% Al	160 Amps.	4.4"/min.	O_2 .01% N_2 or CO .06%	Good bead.
WS 152	Pure Mo	160 Amps.	2.2"/min.	O_2 .01% N_2 or CO 0.1%	Improper speed adjustment - melted through sample.



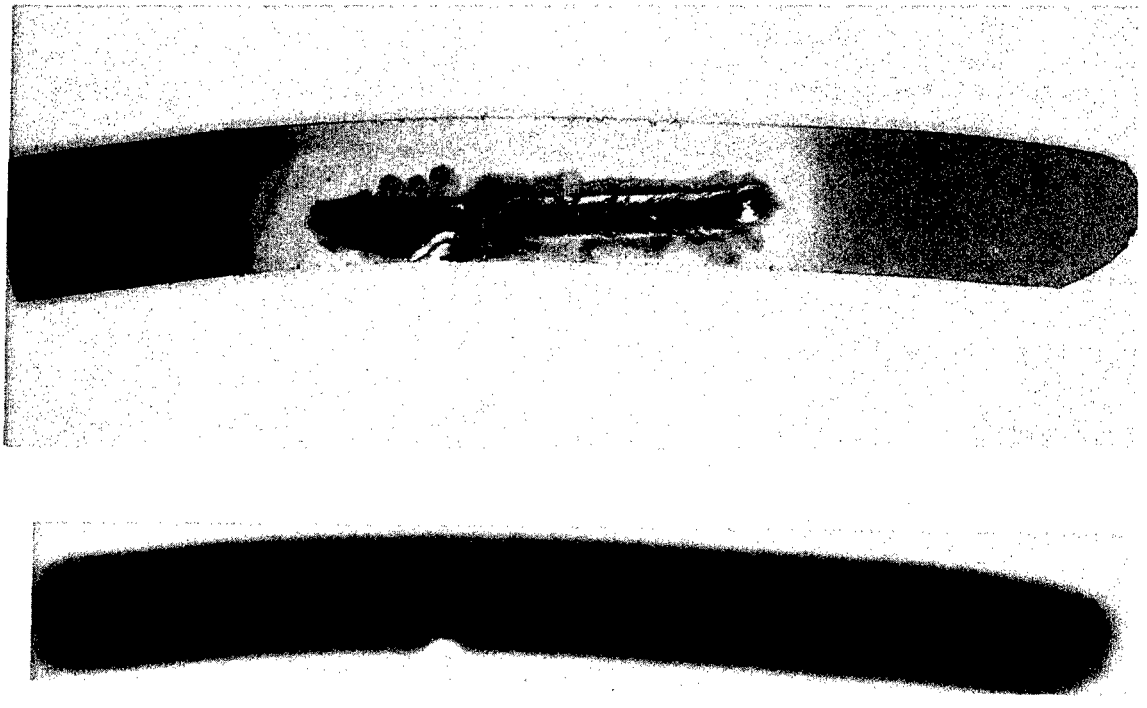
P 53-126-1

FIG. 51 WS 146 VACUUM SINTERED MOLYBDENUM 0.1% C



P 53-126-2

FIG. 52 WS 148, VACUUM SINTERED MOLYBDENUM 0.2% Ti



P 53-136-2

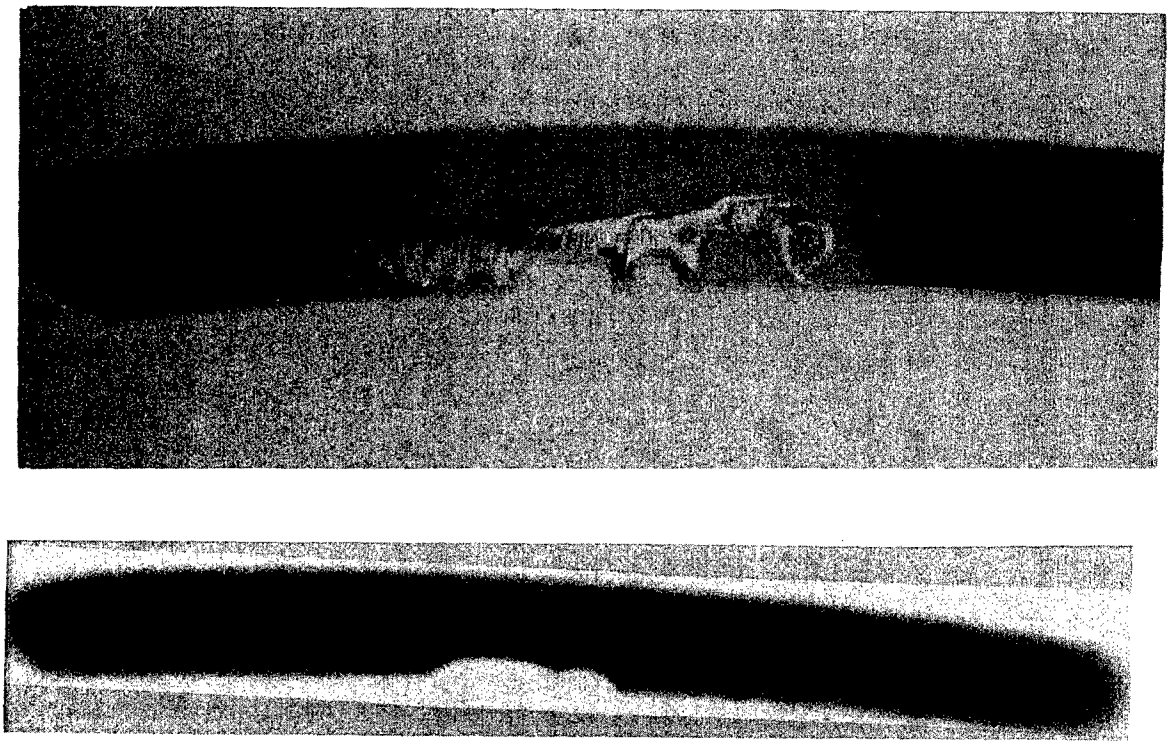
FIG. 53 WS 149, VACUUM SINTERED MOLYBDENUM 0.5% Ti

P 53-126-3

FIG. 54 WS 150, VACUUM SINTERED MOLYBDENUM 0.2% Al

P 53-126-4

FIG. 55 WS 151, VACUUM SINTERED MOLYBDENUM 0.5% Al



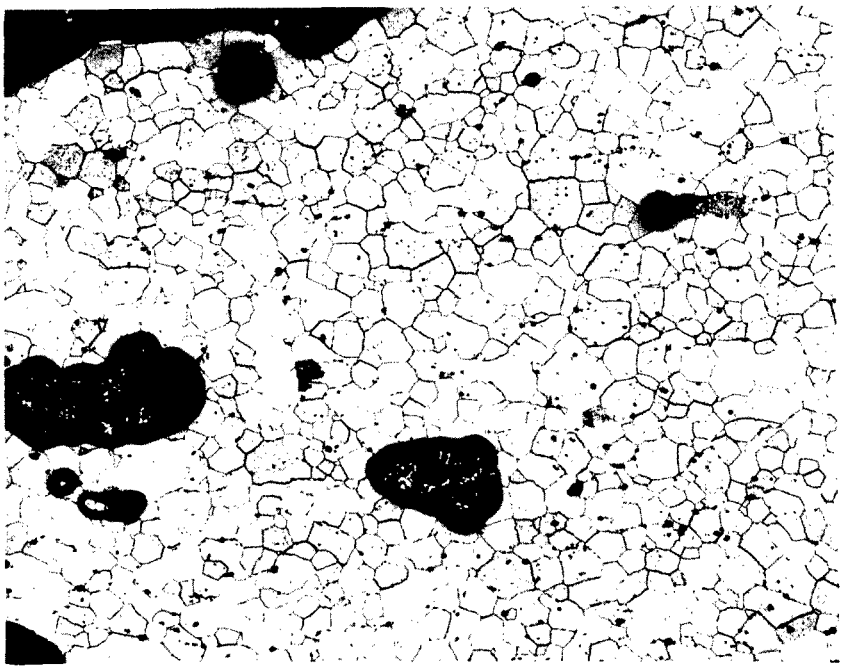
P 53-136-1

FIG. 56 WS 152, VACUUM SINTERED PURE MOLYBDENUM

molybdenum control sample WS152, Figure 56, showed reduced porosity over the commercially sintered material, Figure 29, and only slightly more porosity than the deoxidized vacuum sintered material. This condition indicates that vacuum sintering may have helped reduce weld bead porosity an amount equal to that brought about by using deoxidizers.

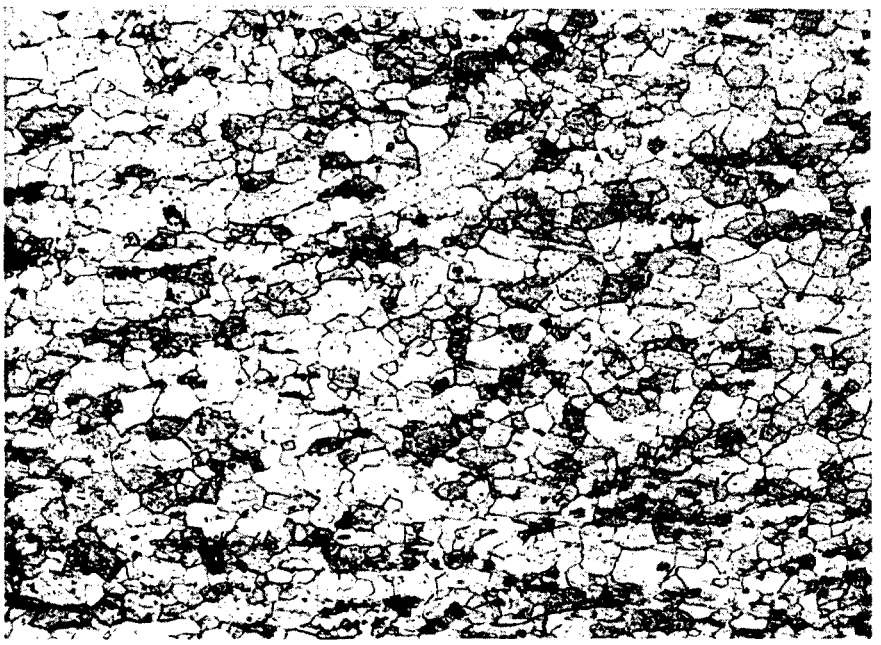
Metallographic sections of the several samples of deoxidized material after welding are shown in Figures 57-63. Sample WS146 which was deoxidized with 0.1% C is shown in Figure 57. The area at the base of the weld metal is shown in Figure 57a. The line of fine porosity at the interface between the fused and unfused zones indicates that either the material was not completely deoxidized or that gases other than oxygen are being driven out by the welding temperatures. Carbides in the grain boundaries are shown in Figure 57b and 57c. Figure 57b shows the condition in the fused portion of the weld while Figure 57c shows the zone just back of the fused zone. Figure 57d shows a transcrystalline crack in the parent metal outside the weld. The transcrystalline crack indicates that for this particular sample the material was sufficiently strong in the grain boundaries so that failure occurred through the grains.

Sample WS147, deoxidized with 0.3% C, is shown in Figure 58. A section of the recrystallized parent metal appears in Figure 58a. Considerable porosity is shown here which did not appear in the unwelded material, Figure 58b. Large quantities of carbides were apparently put out of solution during the welding process as shown in Figure 58c. The eutectic type structure shown in Figure 58c is shown again at 500X in Figure 58d. The excessive number of carbides in this sample indicates that carbon additions of 0.3% were much too large. It had been expected that a considerably larger amount of carbon would be lost during the sintering process. The large carbon carry-over from the



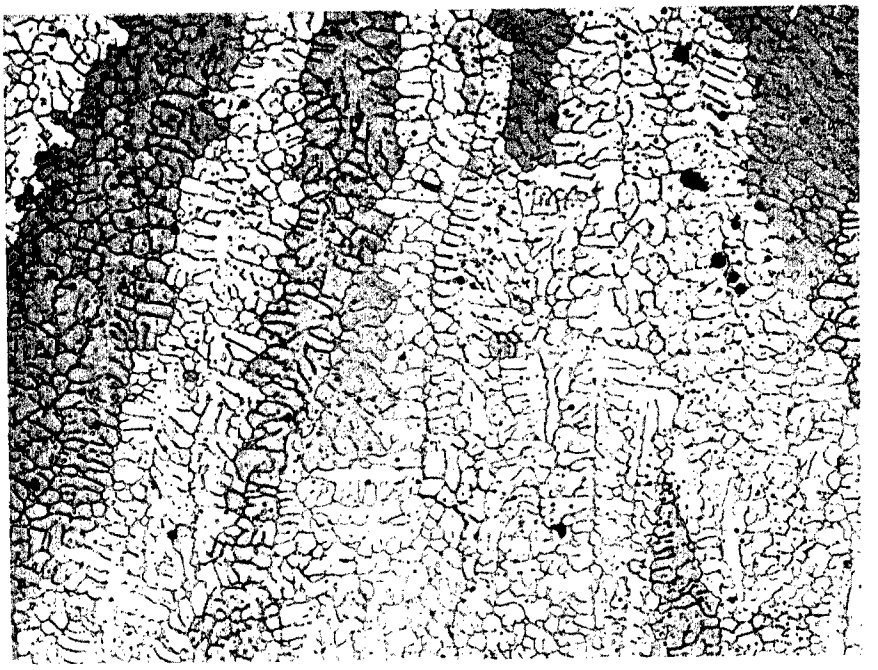
P 53-165-1

FIG. 58a VACUUM SINTERED Mo 0.3% C.
UNWELDED PARENT METAL 100 X



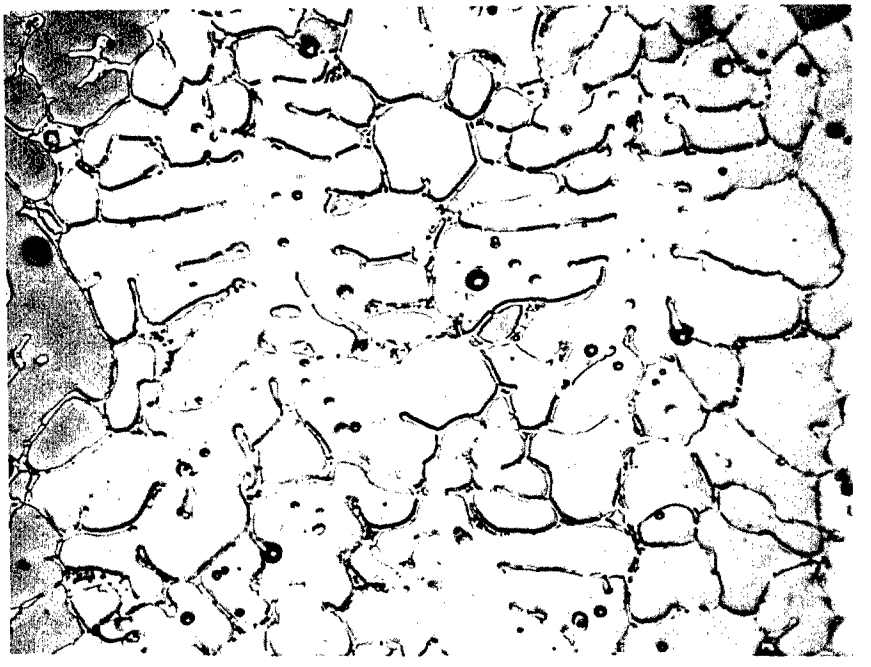
P 53-69-2

FIG. 58b WS 147, VACUUM SINTERED Mo 0.3% C,
PARENT METAL AS RECRYSTALIZED
1200°C 1 HR 200 X



P 53-165-2

FIG. 58 c WS 147, VACUUM SINTERED Mo 0.3% C
CARBIDES IN WELD ZONE. 100 X



P 53-16ɔ-ɔ

FIG. 58 d WS 147, VACUUM SINTERED Mo 0.3% C
CARBIDES IN WELD ZONE 500 X

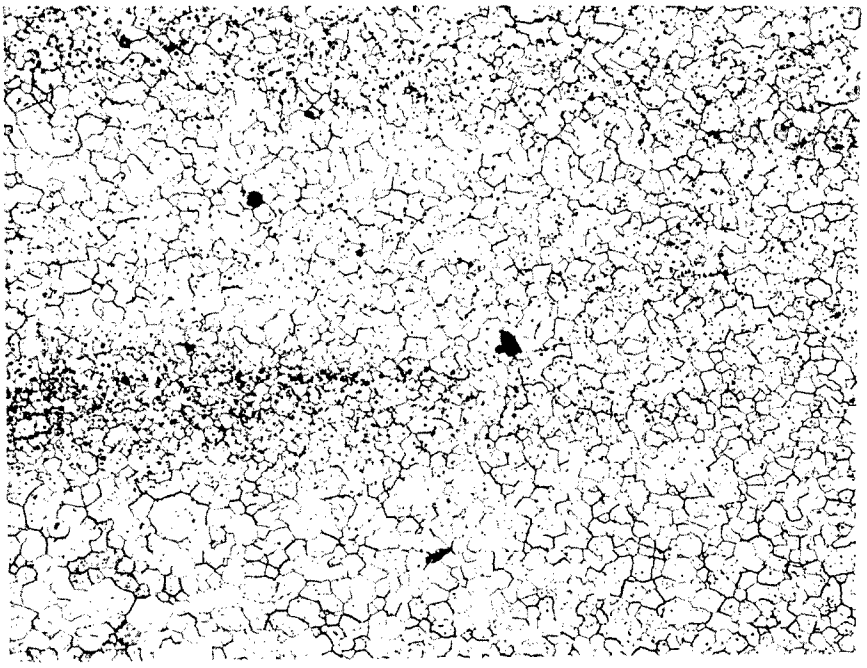


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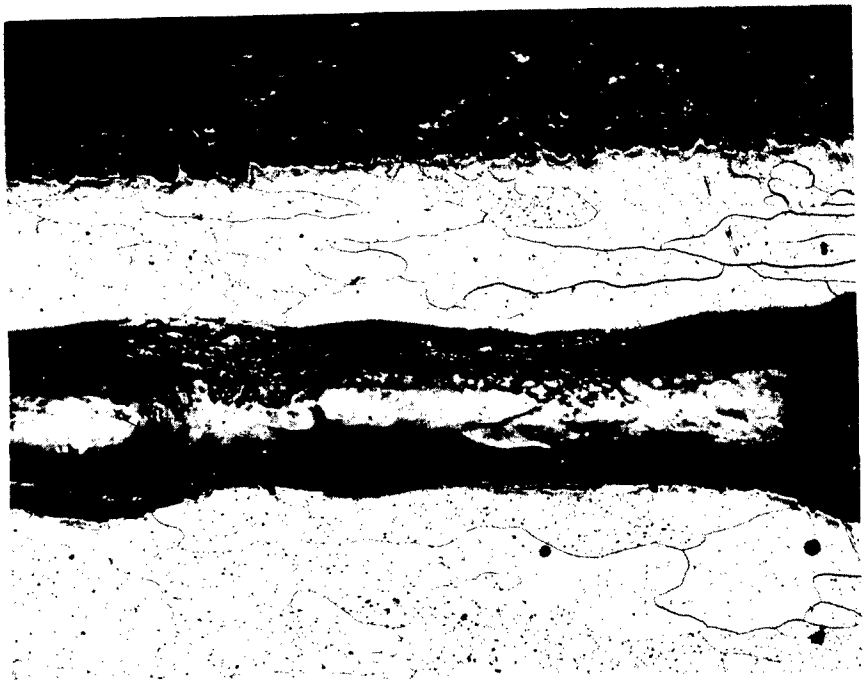
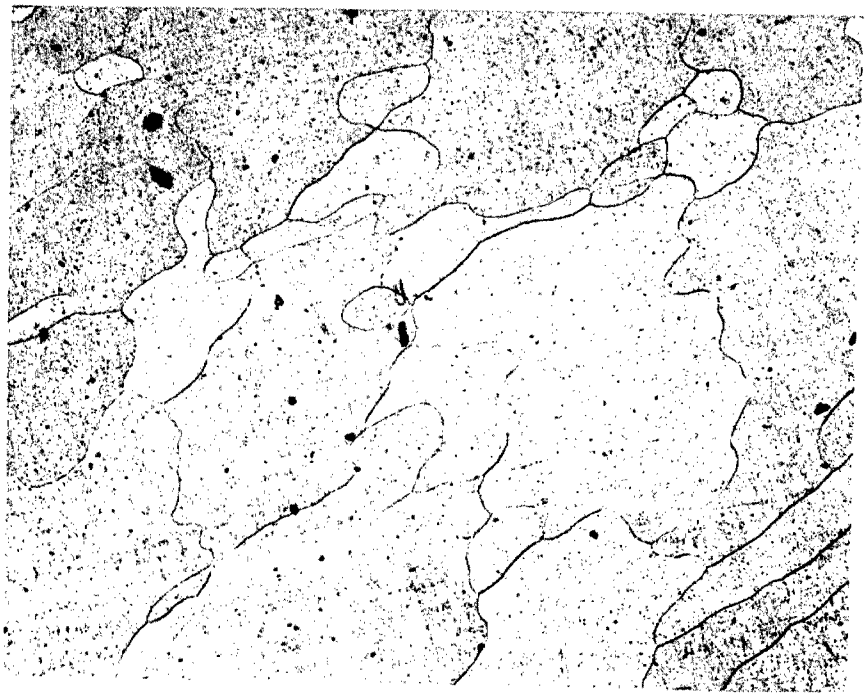
FIG. 59 a WS 148, VACUUM SINTERED Mo 0.2% Ti
UNWELDED METAL. 100 X



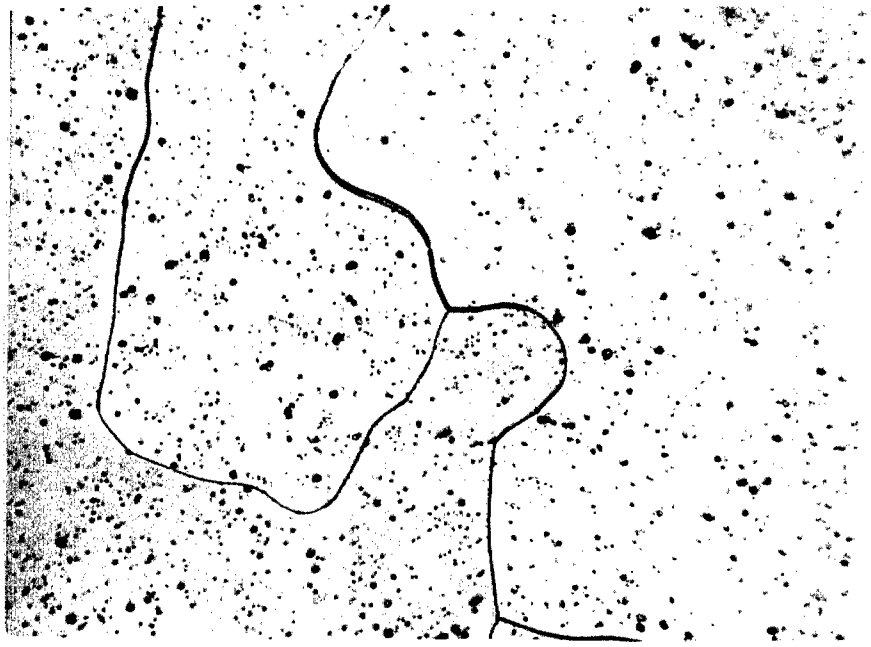
P 53-164-3

FIG. 59 b WS 148, VACUUM SINTERED Mo 0.2% Ti
POROSITY AT BASE OF WELD BEAD,
LONGITUDINAL SECTION 100 X



P 53-164-4

FIG. 59 c WS 148, VACUUM SINTERED Mo 0.2% Ti
WELD ZONE, TRANSVERSE SECTION 100X



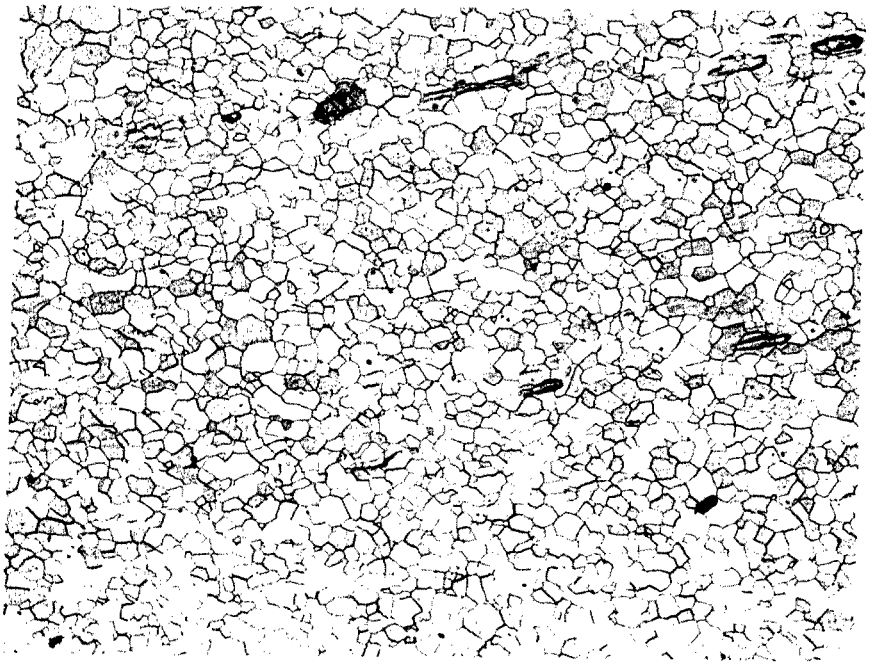
P 53-164-18

FIG. 59 d WS 148, VACUUM SINTERED Mo 0.2% Ti
GRAIN BOUNDARIES IN WELD ZONE,
TRANSVERSE SECTION 500 X

sintering process in conjunction with the formation of porosity during welding indicates that deoxidization during sintering was not complete. Deoxidization occurring during the arc melting of the weld bead could account for the porosity in the material. These preliminary data indicate that carbon may not be a completely successful deoxidizing agent when used in the sintering process.

Samples WS148 and WS149 were deoxidized with titanium. Sections of Sample WS148 deoxidized with 0.2% Ti are shown in Figure 59. The effect of titanium oxides on the molybdenum grain size in the parent metal are shown in Figure 59a. This region appears to be subject to some segregation. Figure 59b shows a long gas hole below the weld bead. It is apparent that some gas was present in the parent metal after deoxidization; either the oxygen was not completely removed or the titanium oxides were broken down or vaporized during welding. Figure 59c shows a transverse section of the weld zone. The irregular shape of the grains is characteristic of transverse sections of weld metal grains when their long axes is roughly parallel to the direction of welding. This condition did not exist in all the samples and the reasons for different orientations are not completely understood. Thermal conditions may alter the grain orientation but other, as yet unknown conditions, also appear to change the grain orientation in the weld beads. Figure 59d shows the grain boundaries of the weld metal. These boundaries appear clean and free from oxides and other foreign materials. The wide grain boundary zone in the center of Figure 59d is only a step effect due to the differential electro-polishing rate on the two grains.

Sample WS149, deoxidized with 0.5% Ti, is shown in Figure 60. The parent metal away from the weld is shown in Figure 60a and the center of the weld bead in 60b. The orientation of the grains is much the same as shown in



P 53-164-6

FIG. 60 a WS 149, VACUUM SINTERED Mo 0.5% Ti
UNWELDED MATERIAL 100 X

P 53-164-7

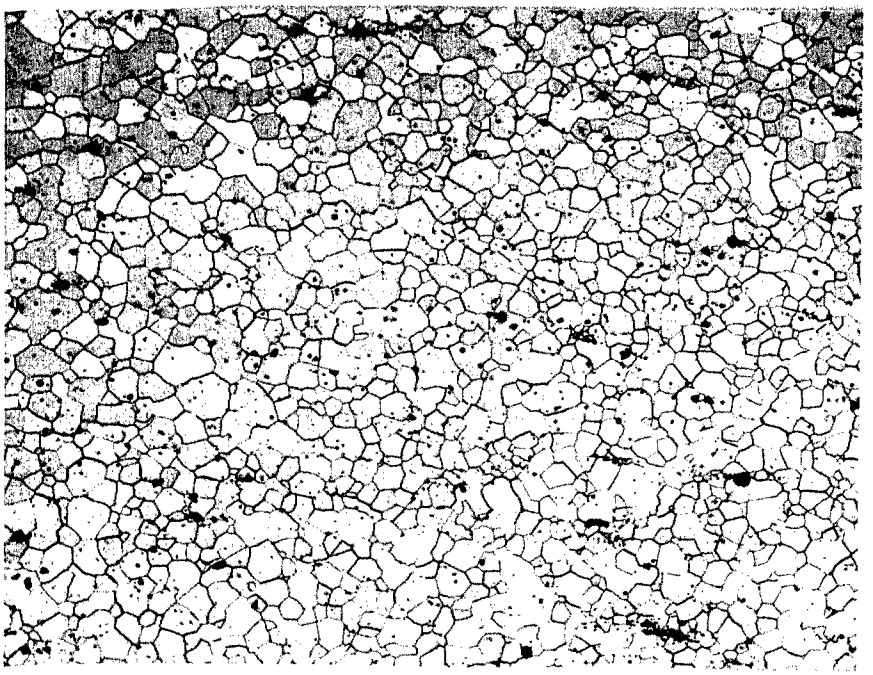
FIG. 60 b WS 149, VACUUM SINTERED Mo 0.5% Ti
WELD ZONE, TRANSVERSE SECTION 100 X

Figure 59C, the relatively fine grains are due to the grain orientation and the transverse direction of sectioning the weld.

The evolution of gas when Ti was used as a deoxidizer indicated that deoxidization during sintering may not have been complete. However, the absence of grain boundary films and the suppression of grain growth through the dispersion of titanium oxides are promising indications. The incomplete deoxidization in Sample WS148 could have been due to incomplete mixing.

Samples WS150 and WS151 were deoxidized with 0.2 and 0.5% aluminum, respectively. The material in WS150 away from the weld zone is shown in Figure 61a; the area at the junction between fused and unfused metal is shown in Figure 61b. The black specks are inclusions and not porosity. The center of the weld zone is shown in Figure 61c. Here again the transverse section cuts the grains perpendicular to their long axes, hence the equiaxed appearance. Sample WS151, Figure 62, deoxidized with 0.5% aluminum showed a different grain orientation from Sample WS150, Figure 61c. The photomicrograph in Figure 62 was made in a longitudinal direction down the center of the weld. The columnar grains extended to the surface of the bead. Their relatively small size should be noted. The absence of porosity at the weld to parent metal junction is also apparent. It is possible that the aluminum not only deoxidized the material but limited the grain size in the weld metal.

A control, Sample WS152, vacuum sintered from pure molybdenum powder without any deoxidizing addition is shown in Figure 63. The parent metal away from the weld is shown in Figure 63a. Apparently the sample recrystallized during welding because the same sample treated earlier at 1200°C for one hour had not completely recrystallized, Figure 63b. The weld zone is shown in Figure 63c.



P 53-164-10

FIG. 61a WS 150, VACUUM SINTERED Mo 0.2% Al
UNWELDED MATERIAL 100 X

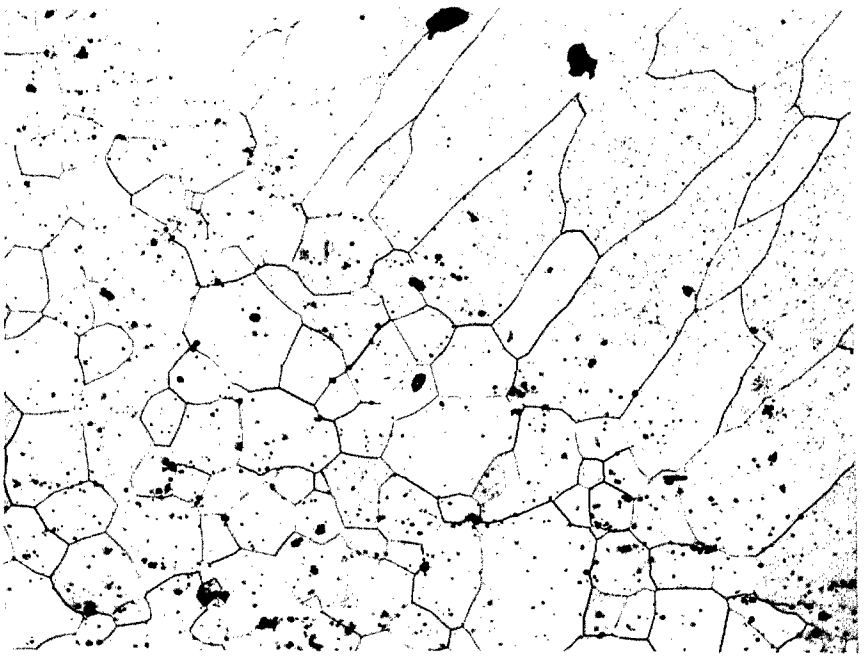


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FIG. 61b WS 150, VACUUM SINTERED Mo 0.2% Al
BASE OF WELD ZONE 100 X

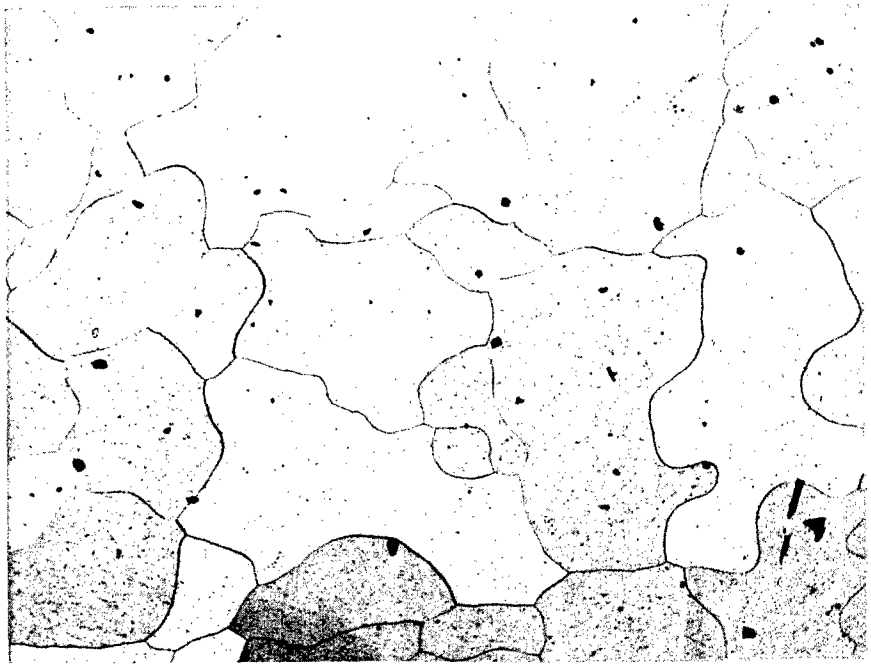

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FIG. 61 c WS 150, VACUUM SINTERED Mo 0.2% A1
TRANSVERSE SECTION OF WELD ZONE.
100 X

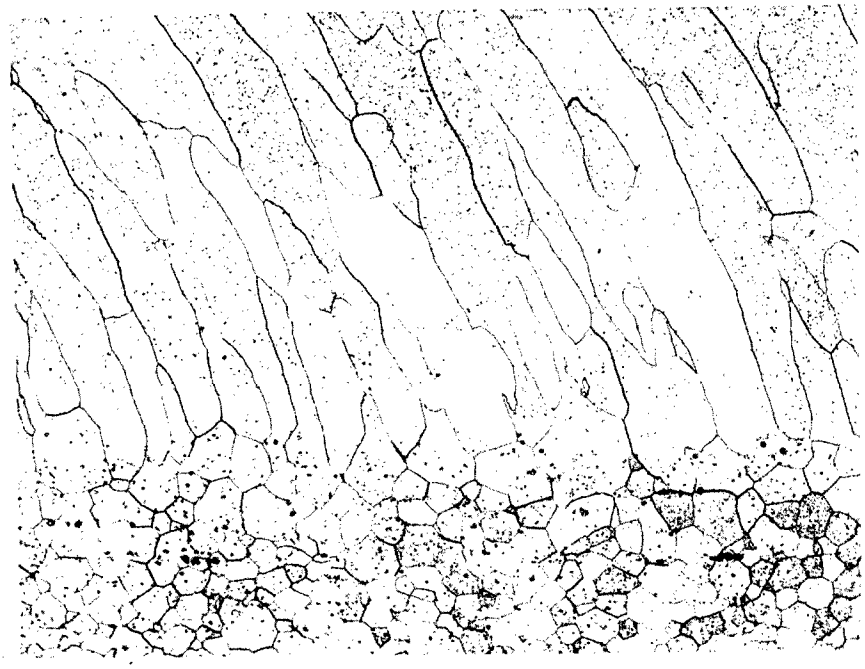

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FIG. 62 WS 151, VACUUM SINTERED Mo 0.5% A1.
BASE OF WELD ZONE. 100 X

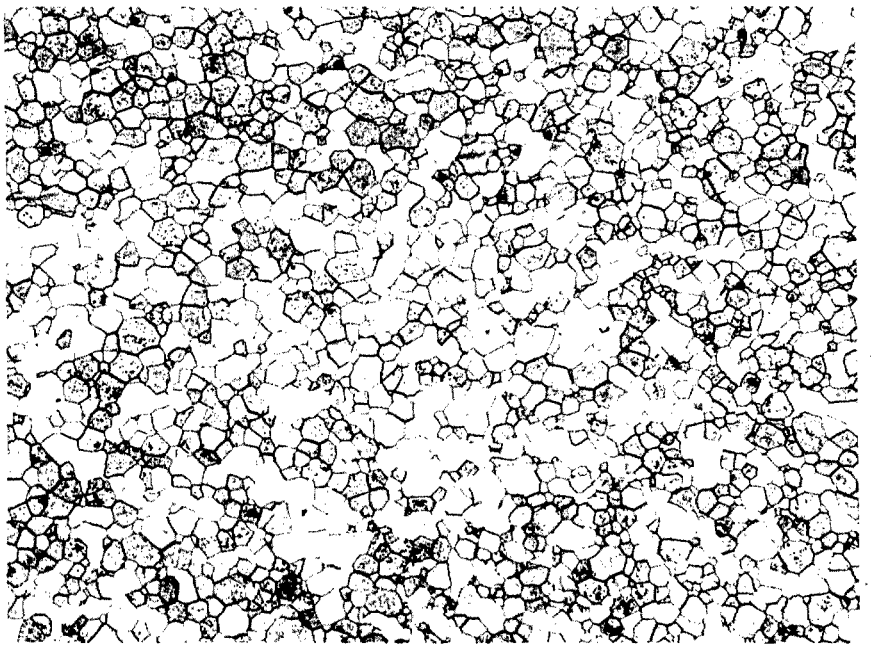

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FIG. 63 a WS 152 PURE MOLYBDENUM, VACUUM SINTERED, UNFUSED MATERIAL 100 X

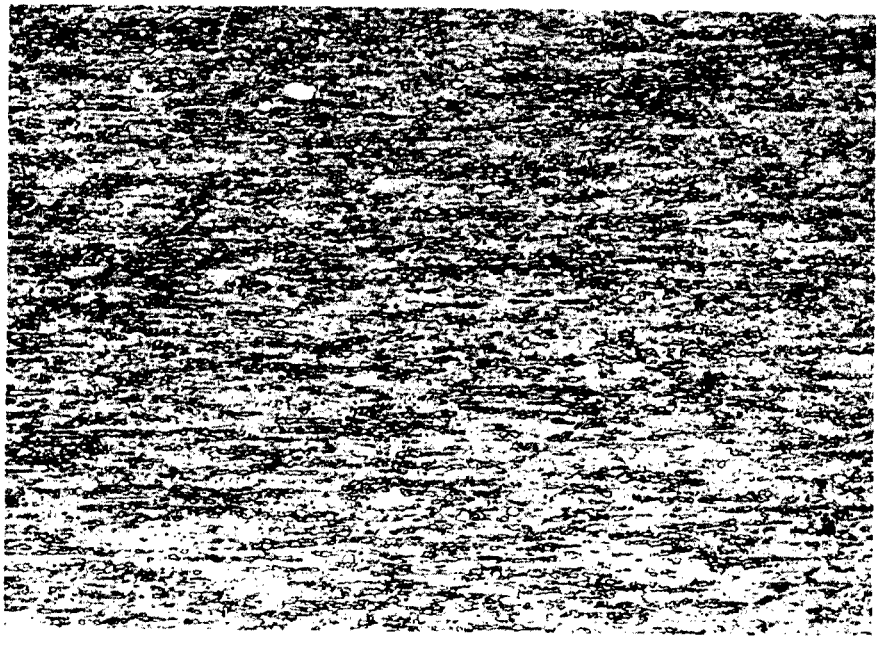

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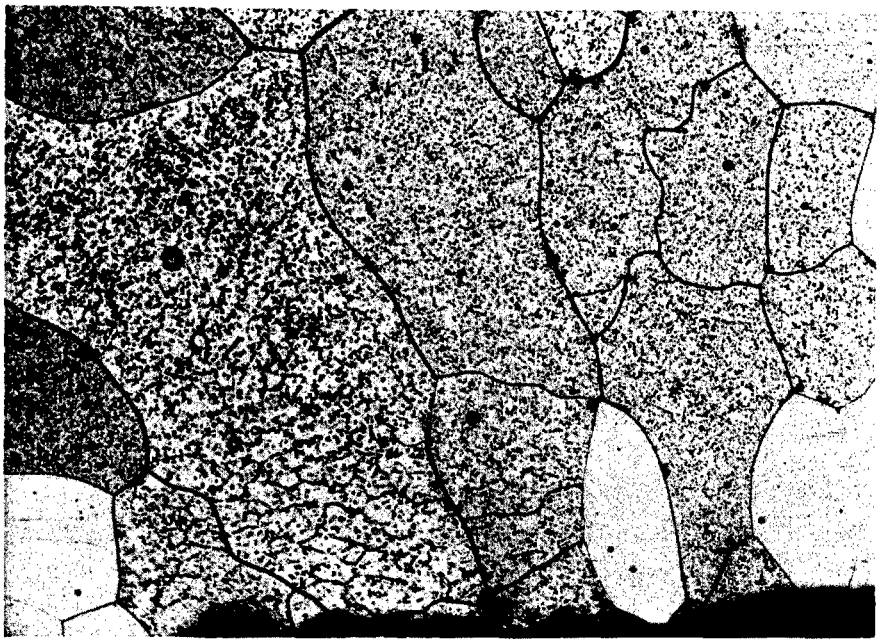
FIG. 63 b WS 152 VACUUM SINTERED PURE Mo BEFORE WELDING, 1200°C 1 HR 200 X



P 53-164-13

FIG. 63 c WS 152 WELD ZONE, VACUUM SINTERED PURE Mo. 100 X

An over-all comparison of the weld metal structures on the basis of the photomicrographs and X-ray pictures shows that, in general, the deoxidizers improve the quality of the weld metal as compared to commercial sintered material. A 0.1% carbon additive, as seen in Figure 57 for Sample WS146, tends to result in porosity at the weld metal to parent metal interface. Excessive carbides result from 0.3% C additions, Figure 58. Titanium produces a good deoxidizing action when used in sufficient amounts, such as 0.5%. However, titanium also tends to increase inclusion content. The presence of a relatively large number of inclusions in the Ti deoxidized material restricted the grain growth in the parent metal and may somewhat reduce growth in the weld metal grains. Titanium deoxidized samples produced a slag-like material on the weld bead surface, Figures 52 and 53. A small amount of the slag was removed and an X-ray diffraction examination of this material showed it to be one of several compounds, Ti_2O_3, Fe_3O_4 or $FeTiO_3$, all of which show similar diffraction patterns. Since the amount of iron present was very small, it is probable that the slag material was Ti_2O_3. This material remained on the sample and apparently was not vaporized.

In general, the least porosity in and around the weld bead was observed in material deoxidized with elements which formed non-volatile oxides. Apparently, when the deoxidization is not completed during sintering, the formation of volatile oxides during welding will produce porosity. Thus, for deoxidization of sintered materials, the investigation of materials forming non-volatile oxides seems in order. However, the exploratory data collected thus far are not conclusive and further work along these lines are planned for future investigations. Other deoxidizers such as Zr, Ta, Cb, Ca, Mg and Mn will also be investigated.

VI - SUMMARY

The data in this report show that molybdenum welds are extremely sensitive to the amount of available oxygen present either in the welding atmosphere or in the parent metal. Oxygen in the welding atmosphere in amounts over 0.1 to 0.2% will cause the formation of hot short cracks in carbon deoxidized arc cast molybdenum. Ductility of weld beads in molybdenum appears to be extremely sensitive to oxygen. The brittle to ductile transition temperature range is rapidly extended upward by the presence of oxygen in the welding atmospheres. The data indicate that, if conventional gas arc shielding is used, efficiencies in shielding must be 99% or better with respect to air if hot short cracking is to be prevented. It should be understood these shielding efficiencies are based on oxygen alone. The effects of nitrogen are as yet undefined but will be investigated in the future.

The data show that the parent material must be deoxidized by some means either during arc casting or sintering if crack and porosity free welds are to be obtained. The exploratory studies indicate that the use of deoxidizers which form oxides stable at temperatures attained in the weld bead are to be preferred over deoxidizers which form volatile oxides provided deoxidization is not completed during sintering.

It should be remembered that the data in these investigations covered only oxygen. It is probable that other gases may cause porosity and cracking. The ductility may also be adversely affected by gases other than oxygen. Nitrogen, hydrogen, carbon-monoxide and carbon-dioxide may also have some deleterious effects.

VII - CONCLUSIONS

1. Deoxidization of sintered molybdenum reduces the amount of porosity in the weld bead and at the parent metal to weld bead interface.
2. Deoxidization of sintered molybdenum may reduce the tendency toward hot short cracking of the material.
3. As yet insufficient data are available to determine the best type of deoxidization practice for making sintered molybdenum.
4. The presence of 0.2% of oxygen in the welding atmosphere is sufficient to cause hot short cracking in weld beads formed in arc cast carbon deoxidized molybdenum.
5. The ductile to brittle transition temperature range of molybdenum is adversely affected by the presence of oxygen in the welding atmosphere.
6. The efficiency of any arc welding gas shielding device such as a hand torch must be at least 99% or hot short center bead cracks will occur.
7. The welding atmosphere and the weld metal must have oxygen reduced to an extremely low level before crack free ductile welds may be produced.
8. Gases other than oxygen should be investigated to determine their effect on the ductile to brittle transition temperature range, porosity and hot short cracking.

VIII - RECOMMENDATIONS

The investigations of the formation of weld beads in molybdenum has demonstrated the need for investigations of several different factors.

1. The effects of oxygen in the welding atmosphere on the ductile to brittle transition temperature of carbon deoxidized arc cast molybdenum in the

range below 0.2% O_2.

2. The effects of nitrogen upon the hot short cracking, porosity, and the ductile to brittle transition temperature.
3. The interrelationship between O_2 and N_2 in the welding atmosphere.
4. The effects, on ductility of the welds, of further purification of the welding atmosphere to eliminate trace O_2 and N_2 from the argon.
5. Investigate more quantitatively the effects of deoxidizers on sintered molybdenum. The following deoxidizers should be tried: C, Ti, Ta, Zr, Cb, Ca, Mg and Mn.
6. Determine the effects of trace elements Fe and Ni upon the welding characteristics of sintered molybdenum.
7. Determine if possible which of the gaseous elements O_2, N_2, or H_2 causes porosity in sintered molybdenum.
8. Study the efficiencies of manual arc shielding methods by means of Schlieren photographs.
9. Determine the effects of cold work in the weld bead upon the ductile to brittle transition temperature range.

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